

# **Drug Enforcement Administration**

## **Office of Forensic Sciences**

**Analysis of Drugs Manual**

**September 2019**

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## **1.0 Quality Assurance**

### **1.1 Scope**

The quality assurance program for drug analysis includes:

- Testing proficiency
- Validating methods
- Verifying and maintaining instruments and equipment
- Verifying and properly handling reference materials (RMs)
- Monitoring storage conditions
- Peer review

### **1.2 Definitions and Abbreviations**

- Terminology used in the Analysis of Drugs Manual (ADM) is defined in Appendix 1A.
- Use of abbreviations is defined in Appendix 1B.

## **2.0 Proficiency Testing Program**

- The Proficiency Testing Program (PTP) consists of four components:
  - Inter-laboratory proficiency testing (PTP)
  - Internal (intra-laboratory) proficiency testing (IPTP)
  - Blind proficiency testing (BPTP)
  - External proficiency testing (EPTP)
- PTP and IPTP samples are prepared by DEA laboratories.
- BPTP samples are prepared by SFL1.
- EPTP samples are obtained from an appropriate outside source. (See Appendix 1H)

### **2.1 Preparing Proficiency Samples**

#### **2.1.1 Preparing Proficiency Samples of Known Composition**

The PTP Coordinator or designee:

- Prepares a proficiency sample of known composition that is representative of exhibits normally encountered. A composite is prepared to allow for at least:
  - PTP: 14.0 g of material for distribution
  - IPTP: 2.0 g of material for distribution
  - BPTP: As needed for composition request
- Uses fully documented reference materials (RMs) to prepare proficiency samples.
- Homogenizes the mixture using techniques appropriate to the sample.
- Records activities on the Forensic Chemist Worksheet (DEA-86) or equivalent, including the following information:
  - Unique identifier of each RM used
  - Weights or balance printouts
  - Sample preparation process, including homogenization, sieving, and mixing procedures, as well as specific equipment used
  - Results of analytical testing, including final purity of the controlled substance(s)

- Calculations (or spreadsheets)
- Evaluation process for sample acceptance or rejection
- Preparer's identification and date
- Analyzes the bulk proficiency sample, prior to distribution.
- Identifies or explains sample components.
- Quantitates controlled substance(s) or listed chemical(s).
- Reviews analytical results for acceptance.
  - Every component is identified or explained.
  - The calculated purity is  $\pm 5\%$ , relative to the expected purity.
- Rejects proficiency samples not meeting acceptance criteria.
  - Reprocesses rejected proficiency sample for acceptance or transfers the rejected material to the destruction coordinator for disposition.

### 2.1.2 Preparing Proficiency Samples from Evidence

The PTP Coordinator or designee:

- Reviews selected evidence for acceptance against the following criteria:
  - A Disposition of Drug Evidence (DEA-48) has been issued and approved by the Laboratory Director (LD) or designee for use in the program. SFL1 may obtain an authorization memo approved by the LD.
  - The original analysis is less than one year old (if possible).
  - A composite (formed in accordance with the DEA Evidence Sampling Plan) exists of at least:
    - PTP: 14.0 g of material available for distribution
    - ITP: 2.0 g of material available for distribution; REDACTED
- NOTE: In the case of tablets or capsules where there is insufficient material in the composite portion for distribution, prepare samples from the bulk material.
- Removes selected evidence from the vault, in accordance with the laboratory's documented chain-of-custody procedures (i.e., LIMS).
- Obtains a gross weight, prior to opening.

- Records the sample separation process, including all weights (or balance printouts), preparer's identification and dates, using either the DEA-86 form (or an equivalent format that documents pertinent information), or in an electronic format.

NOTE: A combination of written and electronic records is acceptable.

- Adds the *Other Notes* test in LIMS to document the removal of samples for PTP/IPTP preparation.
- Separates the composited material from the original packaging.
- Homogenizes the composite using techniques appropriate for the sample type, passes the resulting material through a sieve (60-mesh for PTPs and BPTPs and the same mesh size as the original analyst for IPTPs), mixes thoroughly, and annotates the DEA-86 (or an equivalent format) with the process and equipment used.
- Accepts mixtures that continue to appear homogeneous after processing.
- Seals rejected mixtures in the original PSEE and returns the exhibit to the vault for destruction.
  - Document the reason for rejection in the *Other Notes* test in LIMS.
- Annotates the DEA-48 (in the Remarks section) with the amount of material removed, proficiency designation, date and the preparer's identification.
- Reseals any remaining original evidence material and original packaging into the original container (if possible), and obtains a gross weight.
- Annotates any repackaging in the *Other Notes* test.
- Attaches the DEA-86 (or equivalent format) to the *Other Notes* test.
- Returns the remaining original evidence to the vault for destruction.

## **2.2 Inter-Laboratory Proficiency Test Samples**

The LD or designee:

- Selects, prepares, and distributes an inter-laboratory proficiency test (PTP) sample to laboratories and the Forensic Sciences Instruction Section (SFT).
- Prepares PTP samples according to the following schedule:

Laboratory	Month
<b>Special Testing and Research (SFL1)</b>	February, April, June, August, October, December
<b>Northeast (SFL2)</b>	March, July, November
<b>Mid-Atlantic (SFL3)</b>	February, June, October
<b>Southeast (SFL4)</b>	January, May, September
<b>North Central (SFL5)</b>	April, August, December
<b>South Central (SFL6)</b>	March, July, November
<b>Western (SFL7)</b>	February, June, October
<b>Southwest (SFL8)</b>	January, May, September

- Distributes PTP samples by the fifth business day of the month scheduled.
- Sends an email to the SFT Section Chief or designee the day the PTP sample is distributed, alerting the training facility to expect an inbound delivery.
- Assigns the remaining PTP sample to a Forensic Chemist (FC) in the originating laboratory.

The SFT Section Chief or designee:

- Manages the use of PTP samples received.
- Designates the samples as either proficiency tests for the training staff or for general training purposes.

### **2.2.1 Preparing PTP Samples**

The PTP coordinator or designee:

- Prepares PTP samples of known composition per 1-2.1.1.
- Prepares PTP samples from evidence per 1-2.1.2.

### **2.2.2 Packaging and Distributing of PTP Samples**

The PTP coordinator or designee:

- Labels the PTP vials and PSEEs with the following information:
  - The program name, PTP
  - The last two digits of the fiscal year

- The month
- The laboratory number designator (e.g., PTP-17-01-4 for SFL4, PTP-17-05-8 for SFL8)
- Prepares nine individual PTP samples.
  - Places a 1.0 g portion from the prepared bulk material into a labeled vial.
  - Seals the vial into a labeled PSEE.
- Seals the remaining bulk material into a labeled PSEE (e.g., PTP-17-01-4 source material).
- Distributes the PTP samples.
  - Prepares DEA-12 forms for eight samples to be forwarded to the receiving laboratories.
  - Annotates the DEA-12 with the gross weight of the PSEE and the net weight of the enclosed sample.
  - Uses registered mail (return receipt requested), or an approved commercial carrier to transfer the sample.
  - Sends by the fifth business day of the month scheduled.
- Submits the remaining 1.0 g PTP sample and the reserve source material to the evidence specialist.

### **2.2.3 Analyzing PTP Samples and Reporting Results**

The PTP coordinator or designee:

- Maintains the PTP roster from which a FC is assigned (on a rotating basis) to analyze the PTP sample. SFT assigns an FC on staff to analyze the PTP sample or stockpiles the PTP samples for general training purposes.
- Ensures that the analysis is completed in sufficient time for the results to be posted to the appropriate fiscal year folder on the shared drive by the tenth business day of the succeeding month following sample receipt.
- Posts an electronic PDF of the completed Laboratory Report (DEA-113) and the Case Details Report (CDR) with associated combined instrumental files to the shared drive.

NOTE: The results of PTP samples analyzed at SFT for general training purposes are maintained by SFT, and are not submitted to the originating laboratory.

## 2.2.4 Documenting the PTP

The LD or designee:

- Maintains documentation related to each PTP sample originated and each PTP sample analyzed.
- Documents the PTP analysis in LIMS. The documentation required is displayed in the table below.

Originating Laboratory (PTP of Known Composition)	Originating Laboratory (PTP from Evidence)	Analyzing Laboratory
PTP sample number	PTP sample number	PTP sample number
The unique identifier of each RM used	Copy of original DEA-48, DEA-7, DEA-86, and data	Copy of correspondence from the originating laboratory
The manufacturer and lot # of other components used	Gross weight of the original evidence, prior to breaking the official seal	A completed DEA-113, original worksheets or electronic analytical record, and data
A DEA-86 documenting the sample preparation and distribution, including weights and packaging descriptions	Documentation of the amount of material removed from the original evidence and disposition of PTP samples	Copies of DEA-12s documenting transfers of PTP material
Gross weight of the PTP sample and source material PSEEs, after sealing	Gross weight of the PTP sample and source material PSEEs, after sealing	Copy of the summary report from the originating laboratory
Copies of DEA-12s documenting transfers of PTP material	Documentation of any repackaging and weight of the original evidence, after resealing	Documentation that the analyzing chemist received feedback on the results
Analytical results from each laboratory	Copies of DEA-12s documenting transfers of PTP material	Destruction authorization
Copy of summary report	Analytical results from each laboratory	Final disposition
Destruction authorization	Copy of summary report	
Final disposition	Destruction authorization	
	Final disposition	

#### 2.2.4.1 Evaluating Qualitative PTP Results

The PTP coordinator at the originating laboratory:

- Reviews the results and identifies any qualitative inconsistencies. A qualitative inconsistency exists if any of the following conditions are met:
  - A laboratory reports a controlled substance or adulterant not corroborated by at least one other laboratory.
  - A laboratory does not report a controlled substance identified by at least five other laboratories, including the original analysis.
  - A laboratory does not report an adulterant estimated to be above 1% by at least five other laboratories, including the original analysis.
  - A laboratory does not report a controlled substance known to be present or an adulterant known to be present in a PTP sample of known composition.
- Notifies the Office of Forensic Sciences Quality Assurance Section (SFQ) of any identified inconsistency. (See LOM 71)

#### 2.2.4.2 Evaluating Quantitative Values

The PTP coordinator at the originating laboratory evaluates the PTP results using two tests and identifies any quantitative inconsistencies.

The PTP coordinator:

- Calculates purity values from each laboratory to one decimal place.
- Subjects each value to an “outlier” test to determine whether or not that value will be excluded from the calculation of the experimental mean. The values are tested according to the Extreme Studentized Deviate (ESD) formula

$$T_n = \frac{|x_n - \bar{x}|}{S}$$

where  $T_n$  is the Grubbs statistic,  $x_n$  is the tested value,  $\bar{x}$  is the experimental mean, and  $S$  is the experimental standard deviation (including the tested value).

- Declares an “outlier” when  $T_n$  exceeds 2.13 for  $n=8$  sample observations or 2.21 for  $n=9$  sample observations ( $T_{\text{critical}}$  at 95% confidence, two-sided test).
- Removes the “outlier” from the sample data and recalculates the experimental mean. In the event that there are multiple outliers, the originating laboratory will forward the summary report to SFQ. SFQ will make the final determination whether to include or remove any analytical results before calculating the mean. SFQ will then notify the laboratories of the results.



- Establishes a “target range” for the data set. The target range is

$$\tilde{x} \pm UME$$

where  $\tilde{x}$  the median of the data set and UME is the uncertainty associated with purity determination as calculated using the PTP Summary on the Office of Forensic Sciences Document Control Center (SFDCC).

- Identifies an inconsistency when a quantitative value is determined to be an outlier and falls outside the target range.
- Notifies the submitting laboratory and SFQ of an inconsistency in the quantitative result, as soon as possible.

#### 2.2.4.3 Reporting PTP Results

The PTP coordinator at the originating laboratory:

- Prepares a summary report of the results using the PTP Summary spreadsheet on the SFDCC.
- Posts the summary report to the designated file folder on the shared drive by the fifth business day of the succeeding month following receipt of results.

### 2.3 Internal Proficiency Test Samples

The LD or designee:

- Establishes an Internal Proficiency Testing Program (IPTP) for drug analysis within the laboratory.

NOTE: SFT is exempt from IPTP requirements.

- Assigns one sample each year to each FC on staff that has not completed another proficiency sample (i.e. PTP, BPTP, EPTP).
  - Selects samples pending destruction that have been analyzed within a year, if possible.
  - Selects samples analyzed by different FCs on staff, if possible.

NOTE: Samples reanalyzed for purposes other than proficiency testing (i.e., court, inspection) may be evaluated as part of the IPTP.

#### 2.3.1 Preparing IPTP Samples

The PTP coordinator or designee:

- Prepares IPTP samples of known composition per 1-2.1.1.
- Prepares IPTP samples from evidence per 1-2.1.2.

### 2.3.2 Packaging and Distributing ITP Samples

The PTP coordinator or designee:

- Labels the ITP vials and PSEEs with the following information:
  - The program name, ITP
  - The last two digits of the year
  - A sequential number, (e.g., ITP-17-01, ITP-17-02)
- Places a 1.0 g portion into a labeled vial.
- Seals the remaining bulk source material into a new labeled PSEE (e.g., ITP-17-01 source material).
- Submits the ITP sample and source material to the evidence specialist.

### 2.3.3 Documenting ITP Results

The PTP coordinator:

- Maintains documentation related to each ITP sample originated and each ITP sample analyzed.
- Ensures each file includes the following:
  - The CDR or a copy of the front and back of the DEA-86 for the original analysis
  - A copy of the annotated DEA-48, or an authorization memo for SFL1
  - The ITP sample number
  - The gross weight obtained prior to breaking the official seal on the original evidence used for the ITP source material
  - Documentation of the amount of material removed from the original evidence, and the disposition of this material
  - Documentation of any repackaging, and the gross weight of original evidence after resealing
  - Copies of DEA-12s documenting transfers of ITP material
  - The CDR and analytical data from the ITP analysis
  - Documentation that the analyzing chemist received feedback on the results

### 2.3.4 Evaluating IPTP Results

The PTP coordinator or designee:

- Reviews the results and identifies any analytical inconsistencies. An analytical inconsistency exists if any of the following conditions are met:
  - A controlled substance is reported in only one of the two analyses.
  - An adulterant estimated to be above 1% is not reported.
  - A quantitative discrepancy is identified where the UME range of the average of the two reported quantitative values does not include both individual quantitative values.

For example, the reported quantitative values are 44.2% (original) and 51.6% (IPTP). The average is  $47.9\% \pm 2.9\%$  for a range of 45.0% - 50.8%. Since the individual quantitative values are not within the UME of the average, the comparison will result in an analytical inconsistency.

- Reports results to laboratory management.

### 2.3.5 Reporting IPTP Results

The LD or designee:

- Initiates appropriate follow-up action in a timely manner (in response to an analytical inconsistency), in accordance with the Laboratory Operations Manual (LOM) 71.
- Communicates the results of any follow-up action to SFQ.
- Notifies SFQ (each year), of the successful completion of IPTP samples.
- Records results of analysis and documentation of follow-up action in the laboratory's annual management review.

## 2.4 External Proficiency Test Samples

The LD or designee:

- Ensures laboratory participation in an External Proficiency Testing Program (EPTP) for drug analysis.

NOTE: SFT is exempt from the EPTP requirements.

- Receives feedback from SFQ regarding the EPTP results.

### 2.4.1 Completing an EPTP

The LD or designee:

- Obtains one general chemistry sample each fiscal year from an outside source approved by SFQ and the accrediting body.
- Assigns FCs on a rotating basis to analyze the EPTP sample to meet DEA and test provider requirements.
- Returns the results of the analysis to the test provider within the time limits established by the test provider.
- Authorizes the test provider to release the results to the accrediting body.
- Ensures DEA-required identifications such as adulterants and quantitative results are not reported in the “Comments” section.
- Forwards a complete report of analysis including identified adulterants and quantitation results to SFQ at the same time the results are issued to the test provider.
- Maintains documentation related to each EPTP sample analyzed.
- Ensures each file includes the following:
  - The EPTP sample number
  - The CDR and analytical data from the EPTP analysis
  - The completed vendor’s report
  - The completed DEA-113
  - Documentation that the analyzing chemist received feedback on the results

## **2.5 Laboratory Blind Proficiency Test Samples**

The SFQAM or designee:

- Coordinates with the Office of Inspections (IN) for the submission of blind proficiency samples.

NOTE: SFT is exempt from the blind proficiency test requirements.

### 3.0 Validating Qualitative Instrumental Methods

Qualitative method validation and documentation ensures:

- Objective evidence is maintained demonstrating that the method performs as intended.
- Analysts are aware of any known method limitations that can be adequately addressed through additional testing within the overall analytical scheme.
- Methods are capable of detecting low-level controlled substances and are valid for the identification of all controlled and non-controlled substances included within the validation scope.

NOTE: Validation of qualitative methods is an ongoing process over the lifetime of an instrument. The validity of the method for the identification of compounds not included in the initial validation must be demonstrated upon their first encounter.

A qualitative method includes:

- The technique (separation, confirmation, or hyphenated)
- The specific instrument used
- All associated operating parameters required for the analysis

Method validation is required for the following scenarios:

REDACTED

- Implementation of an external published method

NOTE: Color and precipitate tests and thin-layer chromatography (TLC) published methods are verified in each laboratory via analysis of positive controls. (See 1-11)

- Initial validation of a new laboratory-developed method
- Transfer of an existing method to other instruments
- After modification of method parameters per Appendix 1G

The LD or designee:

- Submits parameters of a new laboratory-developed method to SFQ for review prior to validation.
- Ensures methods are fit for their intended purpose and used as validated.
- Ensures methods are validated prior to use for the identification of controlled and non-controlled substances.

- Ensures validation documentation is updated when new compounds are analyzed using a previously validated method.
- Ensures any *ad hoc* validation procedures are performed per 1-3.5

The FC:

- Validates qualitative methods using laboratory reference materials.

NOTE: Throughout the validation section of policy and procedures, the term “sample” is used in reference to laboratory reference material.

- Validates qualitative methods on instruments that have been verified per 1-6.
- Validates separation methods per 1-3.1.
  - Methods using soft-ionization mass spectrometry with no fragmentation are validated per 1-3.2.1.2.1
- Validates confirmation methods per 1-3.2.
- Validates both the separation and confirmation methods of hyphenated instruments per 1-3.1 and 1-3.2.

NOTE: A confirmatory method is defined by its acquisition parameters. Within a particular instrument, the same mass spectrometer (MS) or infrared detector (IRD) method may be interfaced with different separation methods. The MS or IRD method's name should be traceable to the specific set of acquisition parameters validated. A one-time validation and documentation of the specified MS or IRD acquisition parameters per instrument is sufficient.

- Supplements original validation per 1-3.3.
- Prepares a method validation final report using the appropriate template.
- Documents the instrument identifier (i.e., DEA number).
- Ensures traceability by documenting reference material information including salt form and unique identifier (e.g., laboratory identification number or manufacturer and lot number).
- Documents all solution preparations including solvent(s), concentration(s), derivatization procedure(s) (if used), and filtering technique(s) (if used).
- Documents all limitations of the method.
- Submits a final report and data to the Quality Assurance Specialist (QAS) for review.

The QAS or designee:

- Reviews the data and method validation report for accuracy, completeness, and consistency between instruments, if appropriate.
- Ensures the validation documentation is retained.
- Submits the reviewed report to the Laboratory Quality Assurance Manager (LQAM).

The LQAM or designee:

- Reviews and approves the validation report and makes available for use by analysts.
- Submits the final reports to the Laboratory Document Control Officer (LDCO) for posting on the SFDC.

### **3.1 Validating Qualitative Separation Methods**

The FC:

- Defines the scope of the method as general-purpose or limited-purpose.
- Validates qualitative separation methods - gas chromatography (GC), liquid chromatography (LC), or capillary electrophoresis (CE) - using the following characteristics:
  - Selectivity
  - Repeatability (short-term precision)
  - Reproducibility (long-term precision)

#### **3.1.1 Selectivity**

##### **3.1.1.1 Validation Procedures**

The FC:

- For general-purpose methods, prepares solution(s) containing:
  - Earliest and latest expected eluting compounds
  - Compounds expected to be identified using the method (controlled and non-controlled) and internal standards (if used)

NOTE: Initial validation may include a representative group of compounds. Additional compounds are added as needed to supplement the original validation documentation. (See 1-3.3)

- If practical and available, associated alkaloids and derivatization byproducts

- For limited-purpose methods (e.g., cannabinoids, enantiomer, steroids, etc.), prepares solution(s) containing the target analyte(s) and any potentially related compounds.
- Prepares solution(s) at concentrations appropriate for the technique.
  - For general-purpose methods and any other methods used for the analysis of low-level controlled substances, analyze a 0.5% controlled substance marker compound (e.g., fentanyl) as part of the solution or as a separate test solution.
- Analyzes one injection of the solvent blank (with internal standard, if used) and all test solutions.
- Evaluates the data and calculates the following:
  - Retention/migration time and relative retention/migration time for each compound
  - For isocratic/isothermal LC and GC methods, retention factor ( $k$ ) for earliest eluting compound, where

$$k = \frac{(t_R - t_0)}{t_0},$$

$t_R$  is the retention time of the target analyte, and  $t_0$  is the column's dead time

NOTE:  $k$  value does not need to be calculated for gradient methods

- For isocratic/isothermal LC and GC methods, the theoretical retention time where  $k = 1$  to establish the minimum acceptable retention time of the method
- For gradient methods,  $t_R = 2t_0$  to establish the minimum acceptable retention time of the method
- Peak-to-peak signal-to-noise ( $S/N_{pk-pk}$ ) for each compound

#### 3.1.1.2 Acceptance Criteria

The FC:

- Evaluates the data and accepts the method when the following are met:
  - For general-purpose methods, at minimum, cocaine, methamphetamine, heroin, delta-9-THC, oxycodone, and MDMA (if they are within the scope of the method) are visually separated from each other and from the internal standard (if used).
  - For limited-purpose methods, the tested compounds are visually separated from the target analyte(s) and internal standard (if used).
- Evaluates the data for each compound tested using the following criteria:



NOTE: If acceptable data cannot be achieved for a compound, the method may be accepted as valid for those compounds that have been documented to meet the acceptance criteria.

- A single peak with a clear, non-splitting apex is observed.
- Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
- For isocratic/isothermal LC and GC methods, the first eluting compound is retained with an acceptable retention factor of  $k \geq 1$ .

NOTE: Compounds with  $k < 1$  and that are baseline resolved from the solvent peak may be acceptable provided the earliest eluting compound is tested according to 1-3.1.2 and 1-3.1.3 and is documented to fulfill both the repeatability and reproducibility acceptance criteria.

- For gradient LC and GC methods, the first compound elutes with a retention time equal to or greater than twice the column's dead time (i.e.,  $t_R \geq 2 t_0$ ).

NOTE: Compounds eluting before this value and that are baseline resolved from the solvent peak may be acceptable provided the earliest eluting compound is tested according to 1-3.1.2 and 1-3.1.3 and is documented to fulfill both the repeatability and reproducibility acceptance criteria.

- A minimum  $S/N_{pk-pk} = 3$  is observed, including the 0.5% low-level marker compound.

### 3.1.1.3 Reporting Requirements

The FC:

- Documents the selectivity results using the appropriate validation template.
- Documents the retention/migration time ( $t_R$ ,  $t_m$ ), relative retention/migration time, and  $S/N_{pk-pk}$  for each component analyzed.
- Documents the retention factor ( $k$ ) for the first eluting compound for all isocratic/isothermal methods.
- Documents the minimum acceptable retention time corresponding to  $k = 1$  (for isocratic/isothermal methods) or  $t_R = 2t_0$  (for gradient methods).
- Documents the instrument response (e.g., area counts, abundance) of the 0.5% marker compound.
- Documents all method limitations.

### 3.1.2 Repeatability (Short-term Precision)

#### 3.1.2.1 Validation Procedures

The FC:

- For general-purpose methods, prepares solution(s) containing, at minimum, three compounds plus internal standard (if used). One early-, one middle-, and one late-eluting compound must be evaluated.
- For limited-purpose methods (e.g., cannabinoids, enantiomer, steroids, etc.), prepares solution(s) containing the target analyte(s).
  - If a limited-purpose method is expected to have more than four target analytes, prepare solution(s) containing, at minimum, three compounds plus internal standard (if used). One early-, one middle-, and one late-eluting compound must be evaluated.
- Prepares solution(s) at concentrations appropriate for the technique.
- Analyzes the solution(s) 30 times in a single sequence using the method being validated with a blank prior to the 30 injections.
- Measures the retention/migration time ( $t_R$ ,  $t_m$ ) and the relative retention/migration time for each compound tested.
- Calculates the average retention/migration time and the average relative retention/migration time of all injections for each of the compounds tested.

#### 3.1.2.2 Acceptance Criteria

The FC:

- Evaluates the data for each compound tested using the following criteria:
    - The individual retention/migration times measured are within 0.1 minutes (for GC and LC) or 0.3 minutes (for LC-MS and CE) of the average of all injections.
- OR
- The individual relative retention/migration times measured are within 1% of the average of all injections.

#### 3.1.2.3 Reporting Requirements

The FC:

- Documents the repeatability results using the appropriate validation template.
- Documents the retention/migration time and relative retention/migration time for each compound tested.
- Documents the average retention/migration time and average relative retention/migration time for each compound tested.
- Documents all method limitations.
  - If absolute retention/migration times do not meet acceptance criteria, but relative retention/migration times do meet the criteria, then relative retention/migration times must be used during casework analysis. Conversely, if relative retention/migration times do not meet acceptance criteria, but absolute retention/migration times do meet the criteria, then absolute retention/migration times must be used during casework analysis.
  - If acceptable repeatability cannot be achieved for 30 injections, the method may be accepted as valid for the number of sequential injections that have been documented to meet the acceptance criteria. During casework analysis, positive controls must then be analyzed within the validated number of sequential injections. If the exhibit requires additional injections, a blank must be run prior to the second batch of injections.

### 3.1.3 Reproducibility (Long-term Precision)

NOTE: Reproducibility testing is only required if acceptable repeatability was achieved for 30 injections.

#### 3.1.3.1 Validation Procedures

The FC:

- For general-purpose methods, prepares solution(s) containing, at minimum, three compounds plus internal standard (if used). One early-, one middle-, and one late-eluting compound must be evaluated.
- For limited-purpose methods (e.g., cannabinoids, enantiomer, steroids, etc.), prepares solution(s) containing the target analyte(s).
  - If a limited-purpose method is expected to have more than four target analytes, prepares solution(s) containing, at minimum, three compounds plus internal standard (if used). One early-, one middle-, and one late-eluting compound must be evaluated.
- Prepares solution(s) at concentrations appropriate for the technique.
  - Analyzes the solution(s) a minimum of three times over a six week period using the method being validated with a blank prior to the injection.

- Ensures testing includes week 1 and week 6 and that there is a minimum of 1 week between each reproducibility injection.
- Once selectivity and repeatability testing is complete, but prior to the completion of the reproducibility testing, use of the method along with contemporaneous positive controls (i.e. within 24 hours) is permitted. Upon completion of reproducibility testing, a final validation report is submitted to SFDCC.
- Measures the retention/migration time and relative retention/migration time for each compound analyzed.

#### 3.1.3.2 Acceptance Criteria

The FC:

- Evaluates the data for each compound tested using the following criteria:
    - The individual retention/migration times measured during the six week period are within 0.1 minutes (for GC and LC) or 0.3 minutes (for LC-MS and CE) of the values measured on week 1.
- OR
- The relative retention/migration times measured during the six week period are within 1% of the values measured on week 1.

#### 3.1.3.3 Reporting Requirements

The FC:

- Documents the reproducibility results using the appropriate validation template.
- Documents the retention/migration time and relative retention/migration time for each compound tested.
- Documents all method limitations.
  - If absolute retention/migration times do not meet the acceptance criteria for repeatability, but relative retention/migration times do meet the criteria, relative values must be used for evaluation of reproducibility. Conversely, if relative retention/migration times do not meet acceptance criteria, but absolute retention/migration times do meet the criteria, then absolute retention/migration times must be for evaluation of reproducibility.
  - If acceptable reproducibility cannot be achieved for six consecutive weeks, the method may be accepted as valid for the maximum number of consecutive weeks that have been documented to meet the acceptance criteria. During casework analysis, positive controls must then be analyzed within the limited validated timeframe.

### 3.2 Validating Qualitative Confirmatory Methods

The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

The FC:

- Defines the scope of the method as general-purpose or limited-purpose.
- Validates confirmatory methods - mass spectrometry (MS), infrared (IR) spectroscopy, Raman spectroscopy, or nuclear magnetic resonance (NMR) spectroscopy - using the following characteristics:
  - Accuracy

#### 3.2.1 Accuracy

##### 3.2.1.1 Validation Procedures

##### 3.2.1.1.1 Mass Spectrometry

The FC:

- For general-purpose methods, prepares solution(s) containing compounds expected to be identified using the method (controlled and non-controlled).

NOTE: Initial validation may include a representative group of compounds. Additional compounds will be added as needed to supplement the original validation documentation. (See 1-3.3)

- For limited-purpose methods, prepares solution(s) containing all target analytes.
- Prepares solution(s) at concentrations appropriate for the detector.
- Analyzes the test sample(s) using the method being validated.
- Evaluates the data collected against a verified reference database from SFL1. If a reference database is unavailable, evaluations may be made using a commercial library or published literature spectra.

NOTE: For techniques where there is no SFL1 reference database, commercial library, or published spectra, comparisons to data from at least one other ISO/IEC 17025-accredited laboratory may be used.

##### 3.2.1.1.2 Infrared Spectroscopy

The FC:

- Prepares test sample(s) containing:

- Solid-phase: Cocaine HCl, cocaine base, and methamphetamine HCl, at a minimum
- GC-IR (general-purpose methods): Compounds expected to be identified using the method (controlled and non-controlled) at concentrations appropriate for the detector
- GC-IR (limited-purpose methods): All target analytes at concentrations appropriate for the detector

NOTE: Initial validation may include a representative group of compounds. Additional compounds will be added as needed to supplement the original validation documentation. (See 1-3.3)

- Analyzes the test sample(s) using the method being validated.
- Evaluates the data collected against a verified reference database from SFL1. If a reference database is unavailable, evaluations may be made using a commercial library or published literature spectra.

NOTE: For techniques where there is no SFL1 reference database, commercial library, or published spectra, comparisons to data from at least one other ISO/IEC 17025-accredited laboratory may be used.

#### 3.2.1.1.3 Raman Spectroscopy

The FC:

- Prepares test sample(s) containing cocaine HCl, cocaine base, and methamphetamine HCl, at a minimum

NOTE: Initial validation may include a representative group of compounds. Additional compounds will be added as needed to supplement the original validation documentation. (See 1-3.3)

- Analyzes the test sample(s) using the method being validated.
- Evaluates the data collected against a verified reference database from SFL1. If a reference database is unavailable, evaluations may be made using a commercial library or published literature spectra.

NOTE: For techniques where there is no SFL1 reference database, commercial library, or published spectra, comparisons to data from at least one other ISO/IEC 17025-accredited laboratory may be used.

#### 3.2.1.1.4 Nuclear Magnetic Resonance Spectroscopy

The FC:

- Prepare solution(s) containing compounds expected to be identified using the method at concentrations appropriate for the detector.

NOTE: Initial validation may include a representative group of compounds. Additional compounds will be added as needed to supplement the original validation documentation. (See 1-3.3)

- Analyzes the test sample(s) using the method being validated.
- Evaluates the data collected against a verified reference database from SFL1. If a reference database is unavailable, evaluations may be made using a commercial library or published literature spectra.

NOTE: For techniques where there is no SFL1 reference database, commercial library, or published spectra, comparisons to data from at least one other ISO/IEC 17025-accredited laboratory may be used.

### 3.2.1.2 Acceptance Criteria

The FC:

- Evaluates the data for each compound tested and accepts the results when the following criteria are met.

#### 3.2.1.2.1 Mass Spectrometry

- The overall sample fragmentation pattern (relative ion abundances,  $m/z$  values, and isotopic distributions) corresponds to that of the reference spectrum.
  - Relative ion abundance is measured with respect to the most intense signal in the spectrum.
  - For soft-ionization spectra, relative ion abundances and isotopic distributions that vary from instrument to instrument due to the effects of different instrument types and dissociation conditions (e.g. different mass analyzers, collision gas, collision energy, isolation widths, etc.) are acceptable for comparison.
  - Collision-induced dissociation (CID) data and source fragmentation data are not evaluated against each other.
- The measured  $m/z$  values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum.
  - For EI-MS, if the majority of the sample spectrum is of low abundance, then the spectrum is expanded and re-evaluated against a similarly expanded reference spectrum. Both the full and expanded spectra of both the sample and reference must be shown.
  - For high-resolution MS, the measured  $m/z$  values for prominent ions in the sample spectrum are within 5 ppm of the reference spectrum values.
- The molecular ion (or pseudo-molecular ion) must be observed in the sample fragmentation spectrum if it is observed in the reference spectrum.

- No prominent unexplainable extraneous ions are observed in the sample spectrum.
- For soft-ionization spectra without fragmentation, the pseudo-molecular ion, to include salt or solvent adduct signals, corresponds to the theoretical molecular weight of the compound.

NOTE: Soft-ionization techniques that do not yield fragmentation may be used as separatory techniques.

#### 3.2.1.2.2 Infrared Spectroscopy

- The overall sample spectral pattern (relative peak intensities and wavenumbers) corresponds to that of the reference spectrum.
- The observed wavenumbers for prominent and well-defined signals between 2000  $\text{cm}^{-1}$  and 650  $\text{cm}^{-1}$  in the sample spectrum are within 4  $\text{cm}^{-1}$  of those in the reference spectrum.

NOTE: This correspondence may be demonstrated by displaying the measured wavenumbers on each spectrum or by overlaying the sample and reference spectra.

- The sample spectral pattern between 4000  $\text{cm}^{-1}$  and 2000  $\text{cm}^{-1}$  corresponds to that of the reference spectrum.
- No prominent extraneous signals are observed in the sample spectrum.

#### 3.2.1.2.3 Raman Spectroscopy

- The overall sample spectral pattern (relative peak intensities and Raman shifts) corresponds to that of the reference spectrum.
- The observed Raman shifts for prominent and well-defined signals in the sample spectrum are within 4  $\text{cm}^{-1}$  of those in the reference spectrum.

NOTE: This correspondence may be demonstrated by displaying the measured Raman shifts on each spectrum or by overlaying the sample and reference spectra.

- No prominent extraneous signals are observed in the sample spectrum.

#### 3.2.1.2.4 Nuclear Magnetic Resonance Spectroscopy

- The overall sample spectral pattern (multiplicity, relative signal intensity, and chemical shifts) corresponds to that of the reference spectrum acquired using the same solvent.
- The measured chemical shifts for all signals in the sample spectrum are within 0.2 ppm ( $^1\text{H}$ -NMR (with the exception of labile proton signals) and 2 ppm ( $^{13}\text{C}$ -NMR) of those in the reference spectrum.
  - If the sample spectrum does not meet the acceptance criteria, the sample must be acquired using the same solvent and internal standard as the reference spectrum and re-evaluated.



- For other NMR experiments, acceptance criteria must be established within the laboratory and approved by the LD.
- No unexplainable extraneous signals are observed in the sample spectrum.

#### 3.2.1.3 Reporting Requirements

The FC:

- Documents the accuracy results using the appropriate validation template.
- Documents the reference data (or library) used for spectral comparisons.
- Documents all method limitations.

### 3.3 Supplementing Original Validation Documentation

Continuing validation:

- Ensures a previously validated method is fit for the purpose of analyzing newly encountered compounds
- Is required prior to reporting compounds not included in the original validation
- Documents newly encountered limitations

The FC:

- For separation methods, performs selectivity testing and evaluates the data per 1-3.1.1, and supplements the method validation documentation using the appropriate appendix.
  - If the newly encountered compound becomes the earliest eluting substance on the method and  $k < 1$  or  $t_R < 2t_0$ , perform repeatability and reproducibility testing per 1-3.1.2 and 1-3.1.3.
- For confirmation methods, performs accuracy testing and evaluates the data per 1-3.2.1, and supplements the method validation documentation using the appropriate appendix.
- Submits the data and completed appendix to the QAS for review.

The QAS or designee:

- Reviews and approves the data and method validation appendix for accuracy and completeness.
- Ensures the additional validation documentation is retained and made available for use by analysts.

### **3.4 Validating the Cannabis Analysis Separatory Method**

#### **3.4.1 SFL1 Validation Procedures**

The FC:

- Performs and evaluates selectivity per 1-3.1.1.
- Performs and evaluates repeatability per 1-3.1.2.
  - Calculates the THC:IS ratio for each of the injections and documents the RSD of the 30 injections.
- Performs and evaluates reproducibility per 1-3.1.3.
  - Calculates the THC:IS ratio for each of the injections and documents the RSD over a six week period.
- Evaluates the response (THC:IS ratio) using THC reference material solutions at different concentrations (linear assessment).
- Evaluates the response (THC:IS ratio) using cannabis plant material at different THC concentrations (accuracy assessment).

##### **3.4.1.1 Reporting Requirements**

The FC:

- Documents results using the Qualitative Separation Method Validation – SFL1 Template.
- Documents all method limitations.

#### **3.4.2 Field Laboratory Validation Procedures**

The FC:

- Performs and evaluates selectivity per 1-3.1.1.
- Performs and evaluates repeatability per 1-3.1.2.
  - Calculates the THC:IS ratio for each of the injections and documents the RSD of 30 injections.
- Performs an accuracy assessment by analyzing (single injection) extracts of three cannabis plant material samples at different THC levels and evaluating the THC:IS ratio observed.
  - Accepts accuracy assessment data when:
    - The THC:IS for the low concentration sample is <1

- The THC:IS for the high concentration sample is >1.
- The THC:IS for the middle concentration sample is for informational purposes.

#### 3.4.2.1 Reporting Requirements

The FC:

- Documents results using the Qualitative Separation Method Validation – Field Template.
- Documents all method limitations.

#### 3.4.3 Ruggedness Testing

SFQ:

- To ensure method transferability, subjects the fully developed analytical method to inter-laboratory testing with field-level validation at a minimum of two other laboratories.

NOTE: SFL1 is considered a second laboratory for purposes of field-level validation as long as an instrument other than that used to perform the original validation is utilized.

The FC:

- Accepts results from the field-level validations that meet acceptance criteria as described in 1-3.1.1, 1-3.1.2, and 1-3.4.2.
- If ruggedness testing does not pass the field-level validation acceptance criteria, contacts SFQ to determine course of action.
- Reports the results of the ruggedness testing as attachments to the original SFL1 final report.

### 3.5 *Ad hoc* Validation of Qualitative Instrumental Methods

*Ad hoc* validation allows for the emergency use of a newly developed method intended for on-going use prior to completion of all required validation tests and approval of a full validation package.

The FC:

- Notifies the SC of the need for an *ad hoc* method for analysis.

NOTE: *Ad hoc* validation is not required for methods developed for a one-time use.

- Develops a new general-purpose or limited-purpose method.
- Submits the method parameters to SFQ through the laboratory management chain-of-command.
- Initiates validation of a newly developed separation method per 1-3.1.

- Initiates validation of a newly developed confirmation method per 1-3.2.
- Obtains SC approval to use the new *ad hoc* method for casework.
- Documents approval of the deviation using the *Supervisory Approval* or *Deviation* test.
- Analyzes the unknown sample and contemporaneous positive controls (i.e. within 24 hours) using the new method per 2-5.
- Completes validation and documentation of the new method within 90 days for separation methods or 30 days for confirmation methods.

The SC or designee:

- Approves the use of the newly developed *ad hoc* method for casework.
- Ensures full validation procedures and documentation are completed by the required deadlines.

## 4.0 Developing and Validating Quantitative Separation Methods

### 4.1 Proposing a New Quantitative Separation Method

The LD :

- Requests approval for quantitative method development in accordance with LOM 76.

NOTE: Requests are not required for level 2 – 4 validations.

The FC:

- Includes the following information in the protocol:
  - The controlled substance(s) and listed chemical(s) to be quantitated
  - The instrumental technique
  - The results of a literature search supporting the proposed method, if applicable
  - The internal standard (ISTD) (if used), which is based upon:
    - Availability
    - Solubility
    - Response characteristics
    - Inertness
    - Absence from exhibits containing the analyte of interest
- Develops the method meeting the following requirements:

Minimum Method Development Requirements	Protocol
<b>Method Optimization</b>	Optimize the method based on the development process and results from ruggedness testing.
<b>Solution Solubility and Stability Studies</b>	Evaluate the sample RM and Quality Control (QC) solutions for solubility and resistance to decomposition, to other chemical changes, or to physical degradation.

## 4.2 Validating Quantitative Separation Methods

The FC:

- Validates quantitative separation methods using the following characteristics:
  - Selectivity
  - Linearity
  - Repeatability
  - Accuracy
  - Ruggedness
- Prepares a method validation final report. See the specific requirements for documentation listed in the “Reporting Requirements” section of each characteristic and in the Quantitative Method Validation Final Report – Level 1 or Level 2-4 blank forms.
  - Document RM information including: salt form, purity if applicable, unique identifier, manufacturer’s source, and lot number.
  - Document all solution preparations including weights, balance numbers, solvent extractions (if used), and dilution factors.
  - Document limitations to the method determined during method development and any deviations from the approved protocol.
  - Incorporate results from multiple instruments in one final report for level 2, 3, or 4 validations.
- Prepares one final report per method per instrument.
- Submits the final report through the laboratory management chain-of-command.

The LD:

- Submits the Level 1 final report and associated data for approval in accordance with LOM 76.
- Submit all final reports (levels 1 – 4) to the Office of Forensic Sciences (SF) for posting to the SFDCC.

SF:

- Posts all final reports on the SFDCC.

### 4.3 Levels of Validation for Quantitative Separation Methods

The FC:

- Follows the scenarios described below to determine the minimum characteristics required for method validation:

#### 4.3.1 Level 1

- Scenario: Initial validation of a new fully developed method
- Minimum Required Characteristics:
  - Selectivity
  - Linearity
  - Repeatability
  - Accuracy
  - Ruggedness

#### 4.3.2 Level 2

- Scenario: Transfer of a validated method to another instrument
- Minimum Required Characteristics:
  - Selectivity (critical resolution pairs only)
  - Linearity
  - Quality Control (QC) Check

#### 4.3.3 Level 3

- Scenario: The appearance of a new component, not included in the original selectivity study associated with the method.
- Minimum Required Characteristics: Selectivity (new component alone and with target analyte and internal standard, if used).

#### 4.3.4 Level 4

- Scenario: Appearance of a new pharmaceutical preparation (to include new release formulations or different compositions). Notify SFQ of the receipt of new preparations for timely assignment of level 4 validation.

- Minimum Required Characteristics:
  - Accuracy
  - Selectivity (if additional components are present that were not included in the original selectivity testing)

#### 4.3.5 Validation Scheme Summary

Level	Selectivity	Linearity	Repeatability	Accuracy	Ruggedness	QC Check
1	X	X	X	X	X	
2	X	X				X
3	X					
4	X			X		

#### 4.4 Selectivity

##### 4.4.1 Evaluation Procedures

The FC:

- Prepares and analyzes a solution containing only the target analyte and a solution containing only the internal standard (if used).
- Individually analyzes other controlled substances and adulterants routinely found in exhibits containing the target analyte, as well as any available alkaloids or manufacturing byproducts related to the target analyte.
- Calculates peak resolution and evaluates for any interference.
- Performs the following additional analysis if the calculated resolution between the tested compound peak(s) and the target analyte or internal standard is less than 3:
  - Inject a combined solution of the tested compound, target analyte, and internal standard (if applicable).
  - Vary the amount of target analyte to the amount of tested compound in order to evaluate potential interferences or interactions.



#### 4.4.2 Acceptance Criteria

The FC:

- Examines the chromatography of the peaks associated with the critical pairs for both the target analyte and internal standard to ensure that no excessive peak tailing or peak fronting interferes with the visual resolution of the paired peaks.
- Verifies that peaks are visually baseline resolved from the target analyte and internal standard.
- Ensures the target analyte and internal standard (if used), are resolved ( $R \geq 1.5$ ) from each compound tested. Calculates the resolution using the following equation

$$R = \frac{1.18(t_2 - t_1)}{w_{h(1)} + w_{h(2)}}$$

where  $t_2$ ,  $t_1$  = retention time of the target analyte and tested component and  $w_{h(2)}$ ,  $w_{h(1)}$  = width of the target analyte and tested component at half-height.

#### 4.4.3 Reporting Requirements

The FC:

- Reports the retention time ( $t_R$ ), relative retention time (RRT) relative to the target analyte, and resolution from the target analyte and internal standard.
- Documents any method limitations due to resolution such as co-eluting species, tailing, or fronting compounds, etc.

#### 4.5 Linearity

The linear range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.

##### 4.5.1 Evaluation Procedures

The FC:

- Prepares one high-concentration stock solution of the target analyte using an authenticated (or verified) RM and the appropriate solvent.
- From the stock solution, prepares at least seven RM solutions at different concentration levels, using volumetric or gravimetric dilutions.
- Includes the internal standard in the solvent used for preparing the stock and RM solutions, if required by the method.

- Prepares solutions representing an expanded concentration range for the instrument in use. The following table offers an example of preparation procedures:

Solution	Preparation	Concentration (mg/mL)
1	Stock Solution	4.00
2	8:10 dilution of 1	3.20
3	7:10 dilution of 1	2.80
4	5:10 dilution of 1	2.00
5	4:10 dilution of 1	1.60
6	3:10 dilution of 1	1.20
7	2:10 dilution of 1	0.80
8	1:10 dilution of 1	0.40
9	5:100 dilution of 1	0.20
10	2:100 dilution of 1	0.08
11	1:100 dilution of 1	0.04

- Injects all prepared RM solutions a minimum of two times each, in random concentration order.

#### 4.5.2 Data Evaluation

The FC:

- Performs a linear regression analysis using all tested concentrations.
- Plots instrument response, average area ( $A_{Std}$ ), or average area ratio ( $A_{Std}/A_{ISTD}$ ),  $y$ , as a function of concentration,  $x$ .
- Does not include the origin ( $y = 0$ ).
- Visually inspects the best-fit-plot, documents, and rejects data due to obvious dilution errors, instrument malfunction, etc.
- Annotates the resulting slope ( $m$ ),  $y$ -intercept ( $b$ ), and the correlation coefficient ( $r$ ), where

$$r = \frac{\sum_{i=1}^n X_i Y_i}{\sqrt{(\sum_{i=1}^n X_i^2)(\sum_{i=1}^n Y_i^2)}}$$

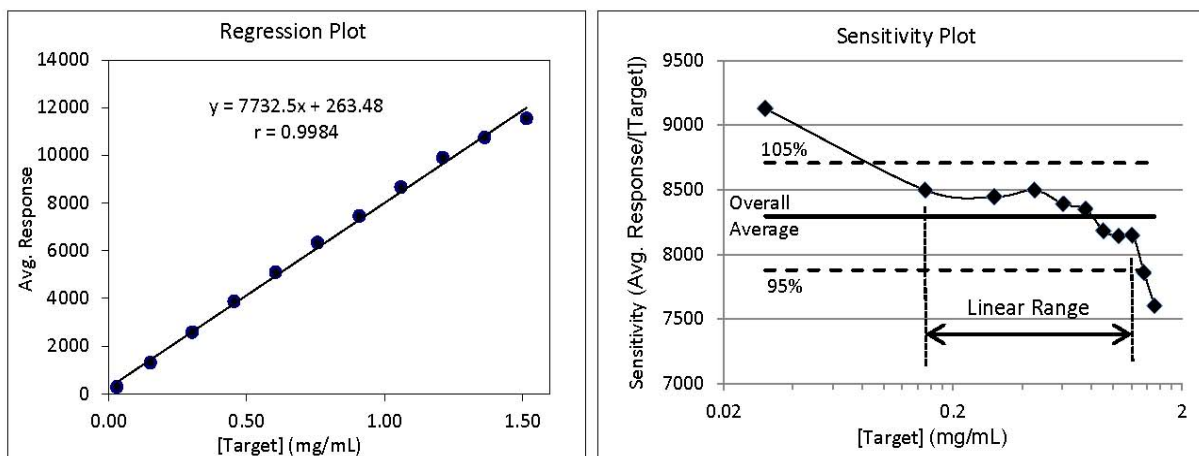
#### 4.5.3 Preparing a Sensitivity Plot

The FC:

- Step 1: Determines the average sensitivity (response/amount) for each concentration analyzed.

- Step 2: Calculates the overall sensitivity across the concentration range tested by averaging the averages obtained in step 1.
- Step 3: Calculates the sensitivity limits by multiplying the overall sensitivity from step 2 by 95% and 105%.
- Step 4: Plots the average sensitivity per concentration, the overall sensitivity average, the 95% limit, and the 105% limit as a function of concentration (logarithmic scale).

#### 4.5.4 Examples of Regression and Sensitivity Plots



#### 4.5.5 Acceptance Criteria

The FC:

- Accepts linearity data for concentrations that fall within the 95 - 105% sensitivity limits.
- Ensures the linear range is determined using at least seven concentrations.
- Based on the sensitivity plot results, if additional linearity solutions are necessary, prepares, runs and includes the data in the evaluation in order to create the widest linear range possible.

#### 4.5.6 Reporting Requirements

The FC:

- Reports each tested solution concentration, average response, and average sensitivity result.
- Reports the linear regression plot with the slope (m), y-intercept (b), and the correlation coefficient (r).
- Reports the sensitivity plot and the established linear range of the method.

## 4.6 Repeatability

The repeatability range of the method is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.

NOTE: Analysts may test/evaluate repeatability and linearity concurrently.

### 4.6.1 Evaluation Procedures

The FC:

- Evaluates repeatability by analyzing at least two solutions representing the low and high end of the linear range.
- Injects each solution five times.
- Measures the instrument response ( $A_{Std}$ ) or area ratio ( $A_{Std}/A_{ISTD}$ ) for each injection.
- Evaluates the repeatability of the method by calculating the relative standard deviation (RSD) of the five injections at each concentration level tested.

### 4.6.2 Acceptance Criteria

The FC:

- Accepts repeatability data that does not exceed 2% calculated RSD for each concentration level tested.
- Calculates the RSD from the sample standard deviation as follows:

$$Std\ Dev = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n - 1}}$$

$$RSD = \left( \frac{Std\ Dev}{Mean} \right) \times 100\%$$

where  $X_i$  is the value obtained from the instrument response ( $A_{Std}$ ) or area ratio ( $A_{Std}/A_{ISTD}$ ),  $n$  is the number of determinations and  $\bar{X}$  is the mean of the values obtained.

- If the RSD for a set of injections does not meet the acceptance criteria, prepares a new solution(s) in order to establish the widest repeatable range that falls within the accepted linear range.

### 4.6.3 Reporting Requirements

The FC:

- Reports the calculated RSD for each concentration level.

- Documents the established repeatability range which fulfills the acceptance requirements.

#### 4.7 Accuracy

The accuracy range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.

##### 4.7.1 Evaluation Procedures

The FC:

- Evaluates the entire linear range during accuracy testing.
- Prepares three mixtures containing a known amount of target analyte at low, middle, and high purity concentrations (e.g., 5%, 50%, and 90%).
  - Prepare the mixtures by combining a known amount of the target analyte with commonly encountered adulterants, diluents, alkaloids, and synthetic byproducts, as applicable.
  - For controlled substances commonly encountered in liquid form, prepare the mixtures by combining the target analyte, adulterants, and diluents into an appropriate solvent or matrix.
  - Factor the documented purity of the authenticated (or verified) RM (target analyte) used to prepare the solid and liquid mixtures into the final solution concentrations.
- From each of the mixtures, prepares three test solutions using the appropriate solvent, so that final solution concentrations of the target analyte represent the low, middle, and high end of the established linear range.
- Prepares new solutions in order to establish the widest accuracy range that falls within the accepted linear range.
  - The following table offers an example of nine accuracy test solutions for an accepted linear range of 0.10 – 1.50 mg/mL:

Solution	Purity (% w/w)	Target Analyte Concentration (mg/mL)
1	5	0.10
2	5	0.75
3	5	1.50
4	50	0.10
5	50	0.75
6	50	1.50
7	90	0.10
8	90	0.75
9	90	1.50

- Quantitates each of the nine solutions using the specified method, and according to 2-6.
- For controlled substances commonly found in pharmaceutical preparations:
  - Uses licit tablets/capsules for the validation, if available.
  - Evaluates the accuracy of the test solutions at low purity values such as 1%, 5%, and 10% for methods intended for quantitation of low dosage pharmaceutical preparations.
  - Ensures all tested formulations (e.g., extended release, different tablet composition, etc.) can be accurately quantitated by the method.
  - Lists formulations that cannot be accurately quantitated as method limitations.
  - Prepares pharmaceutical solutions to ensure the concentration of the target analyte represents the low, middle, and high end of the established linear range.
- For controlled substances that are commonly encountered in multiple salt forms (e.g., cocaine hydrochloride and cocaine base) or mixtures of salt forms, incorporates at least one accuracy sample of the other salt form(s), if available.

#### **4.7.2 Acceptance Criteria**

The FC:

- Accepts accuracy data where the experimentally measured purity (expressed in % w/w) is within  $\pm 5\%$  relative to the known prepared purity.
- If a solution does not meet the acceptance criteria, investigates to determine the root cause and takes appropriate action. This may include preparing and analyzing a new solution to demonstrate the successful range.

#### **4.7.3 Reporting Requirements**

The FC:

- Documents the sample preparation for the solutions prepared as described in 1-4.2.
- Reports the known purity, any correction for RM purity or salt form conversion, the experimentally measured purity, and the percent difference from the known purity value for each solution tested.
- Reports the established accuracy range which fulfills the acceptance requirements.

#### **4.8 Ruggedness Testing**

Ruggedness testing ensures method transferability as described in the Level 1 Protocol for Method Validation.

#### **4.8.1 Evaluation Procedures**

SFQ:

- Subjects the fully developed analytical method to inter-laboratory testing with level 2 validations at a minimum of two other laboratories.

#### **4.8.2 Acceptance Criteria**

The FC:

- Accepts results from the level 2 validations that meet acceptance criteria as described in 1-4.4.2, 1-4.5.5, and 1-4.10.2.
- If ruggedness testing does not pass the level 2 validation acceptance criteria, contacts SFQ to determine course of action.

NOTE: Further method development may be necessary for the level 1 method validation.

- Fully evaluates any further method modifications per level 1 validation criteria.
- Documents all method modifications and deviations from the level 1 protocol in the final report.

#### **4.8.3 Reporting Requirements**

The FC:

- Reports the results of the ruggedness testing as attachments to the level 1 final report.

### **4.9 Working Range**

The working range of the method represents the lowest and highest concentrations tested which fulfill acceptance requirements for linearity, repeatability, and accuracy.

#### **4.9.1 Reporting Requirements**

The FC:

- For a level 1 validation, documents the working range of the method, as described in 1-4.5 through 1-4.7.
- For a level 2 validation, documents the working range of the method incorporating:
  - The working range of the transferred level 1 validated method
  - The instrument specific level 2 validation linear range

- The linear range of a transferred method (level 2 validation) cannot be wider than the working range established during the level 1 validation. The working range of a method is further limited by the level 2 linear range.

#### **4.10 Quality Control Check**

The QC check is an abbreviated evaluation of the repeatability and accuracy of a method.

The FC:

- Uses a QC check when performing a level 2 validation.

##### **4.10.1 Evaluation Procedures**

The FC:

- Prepares two QC solutions at concentrations representing the low and high ends of the linear range.
- Quantitates each QC solution three times using the test method. Measures the instrument response ( $A_{Std}$ ) or ( $A_{Std}/A_{STD}$ ) for each injection.

##### **4.10.2 Acceptance Criteria**

The FC:

- Accepts QC check data that does not exceed 2% RSD of the three measurements.
- Accepts QC check data where the experimentally measured average purity is within  $\pm 5\%$  relative to the known prepared purity.
- If results for the QC solutions are unacceptable, investigates to determine the root cause for any discrepancies and takes appropriate action (e.g., preparation of new QC solutions).

##### **4.10.3 Reporting Requirements**

The FC:

- Reports the calculated RSD for each concentration level.
- Reports the known purity, the experimentally measured average purity, and the % difference from the known purity value for each concentration level.
- Reports the established QC check range which fulfills the acceptance requirements.



#### **4.11 Results Outside of Acceptable Limits**

The FC:

- Does not use any method that fails to meet the required criteria for a level 2, level 3, or level 4 validation, as specified on that instrument.
- Investigates the root cause of the method failure and corrects it.

## 5.0 Validating Quantitative NMR Methods

The FC:

- May develop and validate quantitative NMR (qNMR) methods at one laboratory and transfer the method to another DEA laboratory if the magnetic field strength at the receiving laboratory is equivalent to or higher than the magnetic field strength at the developing, validating laboratory.
- Ensures the following when developing qNMR methods:
  - Analyte signals are clear of interferences.
  - RM and internal standard are soluble in the solvent.
  - A delay between pulses of at least 5 times the spin-lattice relaxation time ( $5 \times T_1$ ) of the signal with the shortest  $T_1$  is used.
  - Solution stability studies are conducted to ensure compounds and internal standard do not react or decompose in the solvent at a rate that will influence accurate quantitation.
  - All qNMR methods incorporate the use of ISTDs.
    - The internal standard in the sample solution is the reference standard.
    - Select the ISTDs based upon availability, solubility, inertness, response characteristics, and absence from exhibits containing the analyte of interest.

### 5.1 Validation of qNMR Method

The FC:

- Validates the qNMR method using instrument and analyte-specific tests.
- Performs instrument tests once for a specific instrument and its probe.
  - Once parameters have been adjusted to produce accurate integrals throughout the spectrum being integrated (normally 0.0-10.0 ppm for proton), save the NMR experiment as the quantitative method to be used for the quantitation of any compound tested.
- Performs analyte-specific tests once for a specific compound using each internal standard/solvent combination to be used.
  - Analyte-specific tests transferred from another laboratory will be documented in the final validation report.

## 5.2 Instrument Tests

### 5.2.1 90° Pulse Width and Spectral Width

#### 5.2.1.1 Evaluation Procedure

The FC:

- Determines the 90° pulse width, using normal calibration procedures.
- Obtains a full spectrum of the qNMR.

#### 5.2.1.2 Acceptance Criteria

The FC:

- Uses pulse widths that are  $\leq 90^\circ$  and  $\leq 10 \mu\text{s}$ .
- Verifies the spectral width covers at least -1 to 11 ppm.

### 5.2.2 Quantitative Spectral Region Uniformity

#### 5.2.2.1 Evaluation Procedure

The FC:

- Prepares a solution containing a compound with one prominent peak (e.g., dimethyl sulfone in chloroform or “doped” D<sub>2</sub>O).
- Conducts the qNMR experiment on the prepared solution by adjusting the NMR parameters, as follows:
  - Set the delay (D1) to at least 5 x T1 of the prominent peak.
  - Set the number of transients to 1 or more.
  - Array transmitter offset (TOF) to move the prominent peak throughout the spectral width with at least 5 equally spaced positions in the region where quantitation will occur (0-10 ppm).
  - Acquire the spectrum.
- Individually phases, drifts, and baseline corrects each spectrum and with the display set to absolute intensity, determines the peak height of the prominent peak for each spectrum.
- Calculates the RSD of these peak heights in the range 0-10 ppm.

#### 5.2.2.2 Acceptance Criteria

The FC:

- Accepts data where the peak height RSD is less than 3%.

#### 5.2.3 Linearity and Accuracy

The FC:

- Performs the linearity study using one common RM.
- Prepares at least 5 different solutions ranging in concentration from 0.1-200%, all containing the same concentration of internal standard.

NOTE: The 0.1% solution will assist the chemist in recognizing the detection limits of the quantitation experiment parameters used.

- Acquires one spectrum for each concentration.
- Integrates the peak groups of the analyte and determines the integral values for each of the concentrations, relative to the integral of the internal standard.
- Plots the results (integral values, y, as a function of concentration, x).
- Calculates the correlation coefficient ( $r$ ). (See 1-4.5.2)
- Calculates the purity of the analyte for each concentration.

#### 5.2.3.2 Acceptance Criteria

The FC:

- Accepts linearity data with a correlation coefficient greater than 0.998.
- Accepts accuracy data where the experimentally measured purity is within  $\pm 5\%$  relative to the known RM purity.
- Ensures that signals above 10:1 S/N ratio and below the probe's analog-to-digital converter overload level (ADC overflow or saturation limit) are tested.

#### 5.2.4 Repeatability

##### 5.2.4.1 Evaluation Procedures

The FC:

- Evaluates repeatability by analyzing at least two concentrations representing the low and high end of the linear range.

- Performs a total of five quantitative experiments for each concentration.
- Calculates the quantitation values (percent purity) for all peak groups of the analyte.

#### 5.2.4.2 Acceptance Criteria

The FC:

- Accepts repeatability data that does not exceed 2% calculated RSD for the following:
  - Quantitative results for the integrals in the individual experiment
  - Quantitative results for the same integral in the spectrum for all experiments for the same sample (e.g., the  $\text{NCH}_3$  of methamphetamine in all five high concentration experiments)

### 5.3 Analyte Specific Tests

#### 5.3.1 Accuracy and Solubility

##### 5.3.1.1 Evaluation Procedures

The FC:

- Performs qNMR experiments on the target analyte RM.
- Integrates peak groups and calculates the purity of the standard at each integral.
- Compares these values to values obtained by authentication data or from purity values from other accepted and validated methods.

##### 5.3.1.2 Acceptance Criteria

The FC:

- Accepts accuracy data where the experimentally measured purity is within  $\pm 5\%$  relative to the known RM purity.

NOTE: Low quantitative values may indicate that the analyte was not fully soluble at that concentration.

- Determines solubility limit by running the target analyte at concentrations until the solubility limits are determined.

### 5.3.2 Analyte Stability

#### 5.3.2.1 Evaluation Procedures

The FC:

- Performs a qNMR experiment on the target analyte solution used for accuracy (1-5.3.1) after 2 hours or longer.
- Compares quantitative results to the original results in 1-5.3.1.2.
- If results increase or decrease over time, determines the rate of change.

NOTE: Decreasing signals are possible due to an exchangeable proton(s).

#### 5.3.2.2 Acceptance Criteria

The FC:

- Uses only integrals where the change in quantitative values is less than 1% per hour in solution.

## **6.0 Instrument Verification and Maintenance**

### **6.1 Procedures and Acceptance Criteria**

The LD or designee:

- Adopts and implements the specific procedures listed below for instruments such as NMR, IR, and Raman.
- For all other instrumentation (i.e., separation components), establishes specific performance verification procedures and acceptance criteria that meet the general system requirements listed below and are consistent with the methods used.

The QAS, Associate Laboratory Director (ALD), and LD:

- Review and approve these procedures before implementation.

### **6.2 Templates**

Performance verification templates aid in the consistent implementation of the following performance verification procedures.

The Instrument Monitor:

- Records specific performance verification procedures established by the laboratory or instrument manufacturer on these templates.
  - For manufacturer-recommended procedures, note the reference citation in the performance verification template.
  - Trace performance verification samples (chemicals, filters, solutions) to a certified source.
- Maintains approved performance verification procedures in each instrument logbook.

### **6.3 Instrument Performance Verification Procedures and Acceptance Criteria**

The Instrument Monitor:

- Performs instrument performance verification according to the procedures detailed in the sections below.
  - Initial or scheduled performance verifications are performed and documented before method validation and before an instrument can be used for case work.

NOTE: Substantial instrument maintenance refers to any non-routine replacement or repair of non-consumable parts and any maintenance performed by non-DEA personnel. (See Appendix 1G).

- For seldom-used instrumentation, the frequency of performance verification procedures stated in this section may be revised by the Laboratory Director. At a minimum, the procedures developed for performance verification intervals shall not be less stringent than the manufacturer's recommendations.

### **6.3.1 Balances**

#### **6.3.1.1 Environment**

The Balance Monitor:

- Ensures that the balance is located on a stable, even horizontal surface and in an area free from exposure to drafts, excessive temperature fluctuations, heat, or humidity.
- Ensures that the balance and surrounding area are orderly and free from sources of contamination.

#### **6.3.1.2 Calibration Check**

- Frequency: Monthly and after substantial maintenance
- Performance Sample: NIST-traceable weights
- Procedure:

The Balance Monitor:

- Inspects and cleans balance prior to performing the calibration checks.
- Uses a clean, soft-haired brush to dust the balance pan, removing any residual material.
- Checks the balance leveling gauge.
- Wears clean, cotton gloves when handling reference weights. For smaller weights, utilizes forceps if a lifting tool is necessary.
  - If available, uses the gloves and forceps that accompany the weights. Metal forceps will never be used as they can scratch the weights.
- Inspects each reference weight to ensure there is no contamination and that they are free of damage.
  - If foreign material is visible, uses a clean, soft-haired brush to remove the material.
  - If there is noticeable damage, removes from service.



- Following manufacturer's instructions, checks the linearity of the balance by using the internal balance adjustment or calibration function.
- Checks the repeatability of the balance by performing three measurements on each of two different NIST-traceable weights.
- When selecting weights for low-mass and high-mass checks, considers the following:
  - Checks should reflect the range of typical sample quantities measured on the balance and should encompass as wide a range as possible/practical.
  - The reference weight tolerance (combined tolerance if more than one weight is used for the high-mass check) should not exceed the accuracy criteria for the balance.
- Tares the balance prior to the initial repeatability check to ensure balance is at zero. Does not tare the balance prior to each subsequent weighing.
- Carefully places the weight on the center of the balance pan, closes balance doors, and waits for the balance reading to stabilize.
- For delta-range balances, considers the balance as two separate balances and checks the repeatability over each range with both high-mass and low-mass reference weights.
- Documents the specific reference weights used (serial number, DEA number, other form of identification) as well as their corresponding conventional masses and class tolerances on the Balance Calibration Check Spreadsheet located on the SFDCC.
- Checks the accuracy of the balance by evaluating each of the three repeatability measurements.
- Maintains calibration check data from balance electronically. Verifies acceptance criteria using the Balance Calibration Check Spreadsheets.
- Acceptance Criteria:
  - Linearity: Verify that the check is successful.
  - Repeatability: The RSD for each set of three measurements will be  $\leq 0.5\%$ .
  - Accuracy: For each reference weight used during repeatability, verify that each of the three individual weights falls within the acceptance range for the corresponding balance readability. Acceptance ranges are based on system-wide reproducibility measurements.

Readability:	Acceptance Range:
0.000001 g	$\pm 0.000010$ g
0.00001 g	$\pm 0.00026$ g
0.0001 g	$\pm 0.0004$ g
0.001 g	$\pm 0.003$ g
0.01 g	$\pm 0.06$ g
0.1 g	$\pm 0.3$ g

- If the measurements obtained on a balance do not meet the acceptance criteria,
  - Repeats the reference weight measurements following the procedures in 6.3.1.2.
  - Contacts service provider if problem is not resolved.
  - Places balance out-of-service.
  - Returns balance to service when the measurements obtained meet the acceptance criteria.

#### 6.3.1.3 Calibration

##### The Balance Monitor:

- Ensures the balances are calibrated annually by an ISO/IEC 17025-accredited calibration laboratory. (See Appendix 1H)
- Ensures the calibration certificates include, at minimum, the following:
  - Balance-specific information (DEA number, manufacturer, model, serial number)
  - Stamp or logo of the accrediting body
  - Calibration certificate identification number
  - Balance capacity
  - Nominal mass
  - True mass (“as found” and “as left” values)
  - Tolerance
  - Uncertainty
  - Adjustments
  - Environmental conditions

- Test equipment used
- Calibration procedures
- Maintains the calibration reports.

#### 6.3.1.4 Reference Weights

The Balance Monitor:

- Utilizes appropriate NIST-traceable weights that are selected based on balance precision and readability. The following are acceptable weight classes based on balance readability:
  - UltraClass – appropriate for balances with 0.00001 g – 0.000001 g readability.
  - OIML R 111 Class E1, E2, F1
    - Class E1 – appropriate for balances with 0.00001 g – 0.000001 g readability.
    - Class E2 – appropriate for balances with 0.0001 g – 0.00001 g readability.
    - Class F1 – appropriate for balances with 0.01 g – 0.001 g readability.
  - ANSI/ASTM E617 Class 1-3
    - Class 1 – appropriate for balances with 0.0001 g – 0.00001 g readability.
    - Class 2 – appropriate for balances with 0.01 g – 0.001 g readability.
    - Class 3 – appropriate for balances with 0.1 g – 0.01 g readability.

NOTE: The weight class used for the annual calibration by an ISO/IEC 17025-accredited calibration laboratory is also appropriate for laboratory use.

- Utilizes reference weights with current calibration certificates.
- For laboratories with more than one set of weights, establishes a mechanism to ensure the weights from one set are clearly distinguishable from those in the other set(s).
- Stores weights in the clean, manufacturer-supplied case under suitable environmental conditions to prevent contamination from dust or other debris and effects from humidity or temperature fluctuations.
- Ensures the weights are not altered in any way (e.g. adding distinguishing marks, mishandling that could cause scratching or chipping).

#### 6.3.1.5 Reference Weight Calibration

The Balance Monitor:

- Ensures the NIST-traceable weights are calibrated once annually by an ISO/IEC 17025-accredited calibration laboratory. (See Appendix 1H)
- Maintains the calibration certificates.

### **6.3.2 Capillary Electrophoresis System**

#### **6.3.2.1 Electrophoresis**

- Frequency: Monthly and after substantial maintenance
- Method: A commonly used method (e.g., general-purpose, limited-purpose, or quantitative)
- Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes): Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.
  - Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Quantitative: Calibrant, low QC, and high QC solutions for the method selected.
- Procedure: Analyze the performance sample one time using the selected analysis method.
- Acceptance Criteria:
  - Tested compounds are visually separated.
  - A single peak with a clear, non-splitting apex is observed for each analyte.
  - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
  - A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.
  - For quantitation methods, criteria listed above and QC solutions are within  $\pm 5\%$  relative to the known prepared purity of the QC sample.

#### **6.3.2.2 Diode Array Detector**

- Frequency: Monthly and after substantial maintenance

- Performance Sample: Not applicable
- Procedure: Perform detector tests and verifications, as recommended by the manufacturer.
- Acceptance Criteria: Test and verification results are within manufacturer's specifications.

### 6.3.3 Gas Chromatography System

#### 6.3.3.1 Chromatography

- Frequency: Monthly and after substantial maintenance
- Method: A commonly used method on each column (e.g., general-purpose, limited-purpose, or quantitative)
- Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes) : Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.
  - Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Quantitative: Calibrant, low QC, and high QC solutions for the method selected.
- Procedure: Analyze the performance sample one time using the selected analysis method.
- Acceptance Criteria:
  - Tested compounds are visually separated.
  - A single peak with a clear, non-splitting apex is observed for each analyte.
  - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
  - The first eluting compound is retained with an acceptable retention factor of  $k \geq 1$  for isocratic/isothermal methods or a retention time  $\geq 2t_0$  for gradient methods.

NOTE: If  $k$  was  $<1$  or  $t_R$  was  $<2t_0$  during validation, the  $t_R$  of the first eluting compound must be within 0.1 minutes of the  $t_R$  obtained during the previous month. Repair events such as column trimming can result in monthly verification parameters that do not meet this

acceptance criterion. For these instances, continue to use the instrument after stating the cause(s) for the discrepancies in the instrument logbook and monitor the reproducibility of the first eluting compound in subsequent months. (See Appendix 1G)

- A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.
- For quantitation methods, criteria listed above, with the exception of the first eluting compound criteria, and QC solutions are within  $\pm 5\%$  relative to the known prepared purity of the QC sample.

#### 6.3.4 Gas Chromatography-Mass Spectrometry System

The FC:

- Completes the performance verification (i.e., tune) of the detector (MS) prior to evaluation of the separation component (GC).

##### 6.3.4.1 Gas Chromatography

- Frequency: Monthly and after substantial maintenance
- Method: A commonly used method on each column (e.g., general-purpose or limited-purpose)
- Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes) : Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.
  - Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
- Procedure: Analyze the performance sample one time using the selected analysis method.
- Acceptance Criteria:
  - Tested compounds are visually separated.
  - A single peak with a clear, non-splitting apex is observed for each analyte.
  - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.

- The first eluting compound is retained with an acceptable retention factor of  $k \geq 1$  for isocratic/isothermal methods or a retention time  $\geq 2t_0$  for gradient methods.

NOTE: If  $k$  was  $<1$  or  $t_R$  was  $<2t_0$  during validation, the  $t_R$  of the first eluting compound must be within 0.1 minutes of the  $t_R$  obtained during the previous month. Repair events such as column trimming can result in monthly verification parameters that do not meet this acceptance criterion. For these instances, continue to use the instrument after stating the cause(s) for the discrepancies in the instrument logbook and monitor the reproducibility of the first eluting compound in subsequent months. (See Appendix 1G)

- A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.

#### 6.3.4.2 Mass Spectrometer Calibration Check

- Frequency: Monthly and after substantial maintenance
- Performance Sample: PFTBA
- Procedure: Perform a standard tune following manufacturer's instructions.
- Acceptance Criteria: Tune results are within the manufacturer's specifications.

#### 6.3.5 Gas Chromatography - Infrared Spectrophotometer

The FC:

- Completes the performance verification of the detector (IR) prior to evaluation of the separation component (GC).

##### 6.3.5.1 Gas Chromatography

- Frequency: Monthly and after substantial maintenance
- Method: A commonly used method on each column (e.g., general-purpose or limited-purpose)
- Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes): Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.
  - Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-,

mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.

- Procedure: Analyze the performance sample one time using the selected analysis method
- Acceptance Criteria:
  - Tested compounds are visually separated.
  - A single peak with a clear, non-splitting apex is observed for each analyte.
  - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
  - The first eluting compound is retained with an acceptable retention factor of  $k \geq 1$  for isocratic/isothermal methods or a retention time  $\geq 2 t_0$  for gradient methods.

NOTE: If  $k$  was  $<1$  or  $t_R$  was  $<2t_0$  during validation, the  $t_R$  of the first eluting compound must be within 0.1 minutes of the  $t_R$  obtained during the previous month. Repair events such as column trimming can result in monthly verification parameters that do not meet this acceptance criterion. For these instances, continue to use the instrument after stating the cause(s) for the discrepancies in the instrument logbook and monitor the reproducibility of the first eluting compound in subsequent months. (See Appendix 1G)

- A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.

#### 6.3.5.2 IR Detector

- Frequency: Monthly and after substantial maintenance
- Performance Sample: A solution containing a semi-volatile substance with a vapor-phase IR spectrum documented in literature.
- Procedure: Analyze the performance sample using an instrument method that collects the infrared spectrum at the flow cell temperature and spectral resolution cited in the reference.
- Acceptance Criteria: Measured peak positions of three high intensity absorption bands are within the experimental resolution of the cited reference values.

### 6.3.6 Infrared Spectrophotometer

#### 6.3.6.1 Transmission Wavelength and Resolution Check

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Polystyrene
- Procedure:
  - Collect polystyrene transmittance spectrum (8 scans;  $4 \text{ cm}^{-1}$  resolution)



- Report the peak positions measured for the following three bands: 3060, 1601, and 1028  $\text{cm}^{-1}$
- Acceptance Criteria: Measured peak positions will be within 4  $\text{cm}^{-1}$  of above-referenced values.

#### 6.3.6.2 Reflectance Wavelength and Resolution Check (for ATR)

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Caffeine
- Procedure:
  - Collect caffeine spectrum (8 scans; 4  $\text{cm}^{-1}$  resolution)
  - Report the peak positions measured for the following three bands: 3111, 1644, and 743  $\text{cm}^{-1}$
- Acceptance Criteria: Measured peak positions are within 4  $\text{cm}^{-1}$  of above referenced values.

#### 6.3.7 Ion Mobility Spectrometer

- Frequency: Monthly and before use at off-site location
- Performance Sample: Manufacturer-recommended compound
- Procedure: Analyze the sample following manufacturer's instructions.
- Acceptance Criteria: Verification tests meet manufacturer's specifications.

#### 6.3.8 Liquid Chromatography System

##### 6.3.8.1 Chromatography

- Frequency: Monthly and after substantial maintenance
- Method: A commonly used method on each column (e.g., general-purpose, limited-purpose, or quantitative)
- Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes) : Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.

- Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Quantitative: Calibrant, low QC, and high QC solutions for the method selected.
  - Procedure: Analyze the performance sample one time using the selected analysis method.
  - Acceptance Criteria:
    - Tested compounds are visually separated.
    - A single peak with a clear, non-splitting apex is observed for each analyte.
    - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
    - The first eluting compound is retained with an acceptable retention factor of  $k \geq 1$  for isocratic/isothermal methods or a retention time  $\geq 2t_0$  for gradient methods.
- NOTE: If  $k$  was  $<1$  or  $t_R$  was  $<2t_0$  during validation, the  $t_R$  of the first eluting compound must be within 0.1 minutes of the  $t_R$  obtained during the previous month.
- A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.
  - For quantitation methods, criteria listed above, with the exception of the first eluting compound criteria, and QC solutions are within  $\pm 5\%$  relative to the known prepared purity of the QC sample.

#### 6.3.8.2 Diode Array Detector

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Not applicable
- Procedure: Perform detector tests and verifications, as recommended by the manufacturer.
- Acceptance Criteria: Test and verification results are within manufacturer's specifications.

#### 6.3.9 Liquid Chromatography–Mass Spectrometry System

The FC:

- Completes the performance verification (i.e., tune) of the detector (MS) prior to evaluation of the separation component (LC).

##### 6.3.9.1 Liquid Chromatography

- Frequency: Monthly and after substantial maintenance

- Method: A commonly used method on each column (e.g., general-purpose or limited-purpose).
- Performance Sample: Prepared based on the method, in an appropriate solvent, with internal standard (if used)
  - General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
  - Limited-purpose (four or less target analytes) : Mixture containing the target analyte(s). Sample will contain a 0.5% low-level marker, if included in the scope of the method.
  - Limited-purpose (more than four target analytes): Mixture containing a minimum of four commonly encountered compounds at known concentrations. Sample will contain an early-, mid-, and late-eluting compound and, if included in the scope of the method, one 0.5% low-level marker. The low-level marker cannot be the early or late-eluting compound.
- Procedure: Analyze the performance sample one time using the selected analysis method.
- Acceptance Criteria:
  - Tested compounds are visually separated.
  - A single peak with a clear, non-splitting apex is observed for each analyte.
  - Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
  - The first eluting compound is retained with an acceptable retention factor of  $k \geq 1$  for isocratic/isothermal methods or a retention time  $\geq 2t_0$  for gradient methods.

NOTE: If  $k$  was  $<1$  or  $t_R$  was  $<2t_0$  during validation, the  $t_R$  of the first eluting compound must be within 0.3 minutes of the  $t_R$  obtained during the previous month.

  - A minimum  $S/N_{pk-pk} = 3$  is observed for each compound tested.

#### 6.3.9.2 Diode Array Detector

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Not applicable
- Procedure: Perform detector tests and verifications, as recommended by the manufacturer.
- Acceptance Criteria: Test and verification results are within the manufacturer's specifications.

#### 6.3.9.3 Mass Spectrometer Tune

- Frequency: Monthly and after substantial maintenance

- Performance Sample: Laboratory-customized or manufacturer-recommended compound
- Procedure: Tune the mass analyzer following manufacturer's instructions.
- Acceptance Criteria: Tune results are within manufacturer's specifications.

#### 6.3.9.4 Mass Spectrometer Calibration

- Frequency: Every 3 months and after substantial maintenance
- Performance Sample: Manufacturer-recommended compound
- Procedure: Calibrate the mass analyzer following the manufacturer's instructions.
- Acceptance Criteria: Calibration results are within the manufacturer's specifications.

### 6.3.10 Nuclear Magnetic Resonance Spectrometer

#### 6.3.10.1 Proton Line Shape

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Chloroform in acetone-d<sub>6</sub> (non-spinning sample)
- Procedure: Obtain <sup>1</sup>H NMR spectrum of performance sample (1 scan, 500 – 1000 Hz spectral width, acquisition time ≥ 8 s).
- Acceptance Criteria: Peak width for chloroform proton signal are equal to or less than 1.0 Hz, 12.0 Hz, and 24.0 Hz at 50%, 0.55%, and 0.11% peak height, respectively.

#### 6.3.10.2 Probe File Update

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Methyl iodide (<sup>13</sup>C-enriched) or other recommended by manufacturer
- Procedure: Tune probe, in accordance with manufacturer's procedures.
- Acceptance Criteria: All 90°-pulse widths for proton and carbon are less than two times manufacturer's installation criteria or 20 μs, or whichever is less.

#### 6.3.10.3 Proton Sensitivity (only for indirect detection probes)

- Frequency: Every six months and after substantial maintenance
- Performance Sample: Ethyl benzene in chloroform-d
- Procedure:

- Obtain  $^1\text{H}$  NMR spectra of performance sample using manufacturer's experimental parameters (e.g.,  $90^\circ$  pulse,  $\text{dl} \geq 60$ , 1 scan,  $\text{lb} = 1.0$ ).

NOTE: The manufacturer's specified sensitivity test may satisfy the proton sensitivity test requirements.

- Record S/N level for the 2.5-7.0 ppm region containing the quartet at 2.65 ppm.
- Acceptance Criteria: Measured S/N value greater than 50% of manufacturer's installation specification is acceptable, but requires further investigation. A value greater than 75% requires no further action.

#### 6.3.11 Polarimeter

- Frequency: Every six months and after substantial maintenance
- Performance Sample: Quartz wave plate filter
- Procedure: Measure the optical rotation of the quartz wave plate at 589.3 nm (sodium D line).
- Acceptance Criteria: The experimentally measured rotation for the quartz wave plate will be within the uncertainty measurement specified in the calibration certificate.

#### 6.3.12 Portable IR or Raman

- Frequency: Monthly and before use at off-site location
- Performance Sample: Manufacturer-recommended compound
- Procedure: Analyzes the sample following manufacturer's instructions.
- Acceptance Criteria: Verification tests meet manufacturer's specifications.

#### 6.3.13 Raman Spectrophotometer

##### 6.3.13.1 Wavelength and Resolution Check (A)

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Polystyrene
- Procedure:
  - Collect spectrum of polystyrene (8 scans;  $4\text{ cm}^{-1}$  resolution).
  - Report the peak positions measured for the following three bands: 3054, 1602, and  $1001\text{ cm}^{-1}$ .
- Acceptance Criteria: Measured peak positions are within  $4\text{ cm}^{-1}$  of above referenced values.

#### 6.3.13.2 Wavelength and Resolution Check (B)

- Frequency: Monthly and after substantial maintenance
- Performance Sample: Caffeine
- Procedure:
  - Collect caffeine spectrum (8 scans; 4 cm<sup>-1</sup> resolution).
  - Report the peak positions measured for the following three bands: 2957, 1328, and 555 cm<sup>-1</sup>.
- Acceptance Criteria: Measured peak positions are within 4 cm<sup>-1</sup> of above-referenced values.

### 6.4 Documentation Requirements

The LD or designee:

- Specifies the format of the instrument logbook.
- Archives the instrument logbook in the laboratory for 75 years.

The Instrument Monitor:

- Maintains records and logbooks for calibration checks, performance verifications, and all other repairs and preventative maintenance.
- Includes in the logbook, at a minimum, the following:
  - The identity of the item of equipment and its software and firmware version
  - The manufacturer's name, type identification, and serial number or other unique identification
  - Evidence of verification that equipment conforms with specified requirements
  - The current location
  - Calibration dates, results of calibrations, adjustments, acceptance criteria, and the due date of the next calibration or the calibration interval
  - Documentation of reference materials, results, acceptance criteria, relevant dates and the period of validity
  - The maintenance plan and maintenance carried out to date, where relevant to the performance of the equipment
  - Details of any damage, malfunction, modification to, or repair of, the equipment

- Ensures that the instrument computer is password protected to control access to the system from unauthorized users.

The FC:

- Documents the completion and all results of the performance verification in the instrument logbook. Includes a copy of the performance verification results, all data, and reports generated in the instrument logbook.
- If data does not meet acceptance criteria, makes modifications to the method according to Appendix 1G or performs routine maintenance to obtain data that meet the acceptance criteria.
  - Includes in the logbook data that does not meet the acceptance criteria.
  - If method modifications are made, analysts must use the method with the updated parameters for sample analysis.
- Documents any problems in the instrument logbook and marks the instrument as out of service if an instrument cannot meet the acceptance criteria for the performance checks after method modifications or routine maintenance.
  - Investigate the nature and cause of any failure, and make the necessary adjustments and repairs to bring the instrument back to operation.
  - If a service call is to be initiated, or if the instrument will be out of service for an extended period of time, clearly label the instrument and corresponding instrument logbook as out of service. Update the logbook monthly until the problem has been resolved and the instrument can meet the specified acceptance criteria.
- Ensures that all instrumental data contains the corresponding DEA identification number, date performed, and initials of the analyst performing the verification.

## 6.5 Instrument Maintenance and Scheduling

The FC:

- Documents maintenance in the instrument logbook.
- For hyphenated techniques (e.g., GC-MS, GC-IR, LC-MS, CE-MS, etc.), properly maintains the chromatographic equipment as well as the confirmatory instrument. Performs necessary maintenance immediately, whenever a problem is identified.
- Performs maintenance of instruments and equipment according to the schedule in Appendix 1C.

#### **6.5.1 Special Procedures**

The LD or designee:

- Develops and documents verification and maintenance procedures for instruments or equipment used outside the permanent facility.



## 7.0 Field Laboratory Reference Materials

The LD:

- Designates a primary reference materials (RM) monitor who will administer the program.
- Designates alternate RM monitor(s).
- Restricts access to significant quantities of RMs.

NOTE: RMs consist of controlled substances, non-controlled substances, precursors, and potentially controlled analogues.

The RM Monitor(s):

- Obtains RMs from SFL1, reputable commercial sources per 1-10.3.2, or another DEA laboratory.
- Ensures quantitative RMs are certified reference materials (CRM).
  - SFL1-produced CRMs have traceable purity values of 98% or greater.
  - SFL1-produced quantitative RMs with a lower purity are used only with LD authorization on a case-by-case basis.
- Treats potentially controlled substance analogue RMs as controlled RMs.
- Follows stock and working RM storage procedures for QC samples.
- Stores, distributes, and accounts for RMs.
- Ensures RMs are verified according to 1-8.
- Retains verification results in either electronic or hard copy format.
- Transfers RMs to another DEA laboratory in accordance with LOM 74.

### 7.1 Stock Reference Materials

- Stock RMs are the laboratory's inventoried stock of RMs which are not readily available to the chemist staff. (See LOM 74)

The RM Monitor(s):

- Classifies stock RMs as quantitative or qualitative materials.
- Lists RMs known to degrade, absorb water in their storage environment, or become otherwise unstable in a bound logbook or in an electronic format. These RMs will not be placed in the working RMs inventory unless proper storage conditions are met (e.g. a desiccator).

## **7.2 Working Reference Materials**

- Working RMs are dispensed from stock RMs that have been verified within the last three years and are made readily available to analysts.

The RM Monitor(s):

- Limits controlled working RM to frequently accessed materials.
- Makes no more than 1.0 g available to analysts as working RMs.
- Classifies and labels controlled working RMs as quantitative or qualitative materials.

## **7.3 Storing Controlled Substances and Listed Chemical Reference Materials**

The RM Monitor(s):

- Stores stock RMs in accordance with LOM 74.
  - Limit access to stock RM containers to the RM monitors and laboratory management, or other personnel designated (in writing) by the LD.
- Stores controlled working RMs in locked containers within a secured access vault per LOM 74.
  - SFL1 stores the working RM collection directly in the in-process vault.
  - Limit access to controlled working RMs to FCs and laboratory management.
- Stores stock and working RMs under appropriate storage conditions per 1-9.

## **7.4 Documentation**

### **7.4.1 Stock Reference Materials**

The RM Monitor(s):

- Maintains records regarding the inventory and transactions of controlled substance RMs in a bound index book or in electronic format to include the following:
  - Name of RM
  - Source of RM
  - Date of receipt
  - Net Weight
    - Record the net weight of the initial stock RM and the remaining amount after portions are removed.

NOTE: Laboratories may use the net weight provided by SFL1 as the initial RM net weight or they may calculate the net weight from the measured gross weight and SFL1 provided tare weight.

- RM Number (a lot number or unique identifier for each RM)
- Transaction reason and date (e.g. transferring to a working vial)
  - If the entire stock RM is provided to the chemist without creating a working vial, the chemist will return the stock RM back to the RM monitor within the same day of transfer.
  - The RM monitor will record the net weight removed, name of recipient and purpose of transfer in the logbook.
  - Removal of more than 1 g of RM requires supervisory approval.
- Final Disposition
- Retains records for a minimum of three years after the RM is consumed that contain at least the following:
  - RM source and lot number or unique identifier
  - Name of analyst performing verification
  - Verification date
  - Verification procedure(s) used
  - Authentication, verification and re-verification data

#### **7.4.2 Working Reference Materials**

The RM Monitor(s):

- Maintains a list of working RMs in a bound logbook or electronic format.
- Maintains a sign-out sheet for non-controlled working RM vials.
- Maintains sign-out sheets for the controlled working RM vials using the Controlled Working RM Log on the SFDCC.
  - A separate log must be used for each RM number.
- Completes an entry in the log when a controlled working RM vial is replenished.
- Monthly, identifies controlled working RMs vials that were not accessed in that time-frame.
- For controlled working RM vials that were not accessed, makes the determination to:

- Remove the controlled working RM vial from the collection.

OR

- Retain the controlled working RM vial in the collection and record the vial weight using, at a minimum, a 3-place balance.
- Immediately notifies laboratory management of any discrepant weights.

NOTE: Weights that differ by more than 0.015 g are considered discrepant. This value was determined from laboratory system-wide measurements and represents the 95% confidence interval.

- For explainable discrepancies, state the cause in the comments section of the Controlled Working RM Log.

The FC:

- Completes the Controlled Working RM Log at the time the material is accessed and returned.
- Uses, at a minimum, a 3-place balance to record vial weights.
- Maintains possession of the controlled working RM vial.
  - The controlled working RM vial cannot be transferred to another analyst before returning the vial.
- Returns the controlled working RM vial within the same business day of receipt.
- Immediately notifies laboratory management of any discrepant weights.

NOTE: Weights that differ by more than 0.015 g are considered discrepant. This value was determined from laboratory system-wide measurements and represents the 95% confidence interval.

- For explainable discrepancies, state the cause in the comments section of the Controlled Working RM Log.

The LD or designee:

- Performs a monthly review of the Controlled Working RM Log to identify potential anomalies.

NOTE: A monthly inventory of the controlled working RMs is not required.

## 8.0 Verifying Field Laboratory Reference Materials

- Verification is the process by which the identity and purity of an authenticated RM is assessed to determine if changes in the RM have occurred (e.g., degradation).
- Verifying RMs will not result in revised purity values. (See 1-8.2)

The FC:

- Verifies all RMs used for identification or quantitation in a DEA laboratory report prior to initial use.
- Re-verifies RMs every three years.
  - If it has been more than three years since the re-verification of a RM, verify the RM prior to use.

## 8.1 Verifying Qualitative Reference Materials

The FC or designee:

- At a minimum, performs a confirmation test and a separation test using appropriate techniques for the compound.  
  
NOTE: Hyphenated techniques may be used for this purpose (GC-MS, LC-MS, GC-IRD, ESI-MS/MS, DESI-MS/MS, or DART-MS/MS, etc.).
- At a minimum, verifies the salt form for cocaine HCl, cocaine base, and methamphetamine HCl.
  - Subsequent verifications of salt form are not required after the initial verification.
- Verifies optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., methamphetamine).
  - Subsequent verifications of optical isomeric form are not required after the initial verification.
- Analyzes the collected data.
  - Compare the spectrum to authentication, previous verification, or literature data.
  - Identify and explain any unexpected components.
- Determines if the RM meets the acceptance criteria.
  - Acceptance Criteria: Data obtained is consistent with authentication, previous verification, or literature data, and does not indicate any significant changes in the composition of the RM.
- Submits verification data and associated documents to a RM monitor for review.

- Places RMs that meet the acceptance criteria in the RM inventory.
- Examines the data for RMs that do not meet the acceptance criteria and chooses the appropriate course of action:
  - Dry the RM, re-verify, and store under an appropriate condition. Update the new storage requirements in a bound logbook or in electronic format. (See 1-7.1)
  - Remove the RM from the inventory and send to SFL1 for purification and authentication.
  - Conduct further analysis including quantitation.
  - Transfer the RM to the destruction coordinator.

## 8.2 Verifying Quantitative Reference Materials

The FC:

- At a minimum, performs a qualitative verification per 1-8.1.  
  
NOTE: Upon initial receipt of a quantitative RM, the reported purity value may be accepted without any additional testing.
- At a minimum, performs a qualitative verification per 1-8.1 and a qNMR for the re-verification of a quantitative reference material.
  - The re-verification date for a quantitative RM is three years from the date the purity was last tested.

NOTE: NMR may be used to satisfy the requirement of a confirmation test.

- qNMR: Quantitate at least one portion of the RM.
  - Inspect the qNMR solution visually to ensure the absence of any insoluble material.
- If qNMR is unavailable for use, performs separation area percent purity (SAPP) test using appropriate methods for the compound.
  - SAPP: Analyze at least one portion of the RM on a quantitative separation technique (GC-FID, GC-MS, CE, HPLC, or UPLC).
- Analyzes the collected data.
  - qNMR: Calculate the purity from an average of at least three integrals.
  - SAPP: Measure peak areas in the chromatogram and divide the compound peak area by the total area of all peaks to obtain an area percent purity.

- GC-FID or GC-MS: Do not include the area of the solvent peak in the determination of the total area of all peaks. This technique assumes that all detected peaks have the same response factor. This technique may not detect all substances present in the RM (e.g., water, solvents, inorganic materials, or thermally labile compounds).
  - LC-UV or CE-UV: If the UV spectra of all peaks are not similar, the area percent purity cannot be calculated. In this case, only make note of the number of peaks present in the chromatogram. If the UV spectra are similar, use the most sensitive UV wavelength. These techniques do not detect substances such as water, solvents, inorganic materials, or compounds with weak or no chromophores.
- Determines if the RM meets the acceptance criteria.
    - qNMR: Purity verification is acceptable when the purity from the qNMR experiment is within the laboratory system's UME of the SFL1 or manufacturer reported purity.
    - SAPP: The area percent purity is  $\geq 98\%$  and agrees with the authentication or previous verification data. The component(s) present in the separation agree with the authentication data or are explainable.
  - Submits verification data and associated documents to a RM monitor for review.
  - Places RMs that meet the acceptance criteria in the RM inventory.
  - Examines the data for RMs that do not meet the acceptance criteria and chooses the appropriate course of action:
    - Perform additional experiments to establish that earlier results were outliers (e.g., error in sample preparation).
    - Dry the RM, re-verify, and store in an appropriate location. Update the new storage requirements in a bound logbook or electronic format. (See 1-7.1)
    - Remove the RM from the inventory and send to SFL1 for purification and authentication.
    - Transfer the RM to the destruction coordinator.

### 8.3 Verifying QC Samples

The FC:

- At a minimum, performs a confirmation test using an appropriate technique for the compound.
- Ensures the data is consistent with stated target analyte of the QC sample.

NOTE: Quantitative verification is performed during the quantitative analysis process. (See 2-6)

#### 8.4 Verifying NMR Internal Standards

The FC:

- Obtains a certified internal standard (IS) with traceable purity from an ISO 17034 accredited commercial source.
- Verifies the IS using one confirmatory test.

NOTE: The reported purity value may be accepted without any additional testing.

- Retains data in a logbook or in an electronic format.
- Disposes of expired IS.

#### 8.5 Documentation

The RM Monitor(s):

- Creates and maintains files for each drug RM to include at least the following:
  - Completed Reference Material Verification Summary Sheet
  - Authentication data provided by SFL1, commercial source, or other DEA laboratory, if applicable
  - Laboratory-generated verification and re-verification data
- Retains files for a minimum of three years from the date the RM is consumed.



## 9.0 Monitoring Storage Conditions

### 9.1 Refrigerators and Freezers

Laboratory Staff:

- Upon receipt of evidence, chemicals, reagents, or RMs, reviews the item to determine if a recommendation for storage conditions is provided or known.

NOTE: Refer to LOM 78 for the storage of hazardous chemicals.

- Labels the item appropriately to ensure proper storage conditions if storage is required at a temperature other than room temperature.
- Places the item in a proper storage device.
- Ensures evidence, chemicals, reagents, or RMs, when accessed for use, are stored under proper conditions.

The Monitor:

- Checks the temperature of refrigerators and freezers used for storing evidence, chemicals, reagents, and RMs which require storage at a temperature other than room temperature.
- Uses a thermometer (appropriate for the required temperature range) to monitor the refrigerators and freezers, according to the criteria shown below.
  - Refrigerator:  $>0^{\circ}\text{C}$  -  $10^{\circ}\text{C}$  ( $32^{\circ}\text{F}$  -  $50^{\circ}\text{F}$ )
  - Freezer:  $\leq 0^{\circ}\text{C}$  ( $32^{\circ}\text{F}$ )

NOTE: A thermometer that enables the laboratory staff to record the temperature without opening the door is preferable.

- Documents the temperature of each refrigerator or freezer on the Temperature Log form on the SFDCC at least once each week.
- Ensures that each storage device is compliant with the listed environmental conditions.
- If a storage device is not compliant with the listed environmental conditions:
  - Posts an "Out of Service" sign on the affected equipment, notifying users not to use the equipment.
  - Evaluates the extent of a problem, and determines the cause and resolves the problem, as soon as possible.
  - Immediately informs a laboratory manager if a storage device is not within the designated parameters

- In situations when evidence storage conditions have been affected by equipment failure, notifies the LQAM immediately to determine a course of action.
- Documents notifications regarding storage conditions monitoring on the Temperature Log form, as well as in the appropriate log (e.g., Standards Log).

The LQAM:

- Determines the appropriate course of action upon notification that the storage conditions have fallen outside the parameters listed above.
- Determines whether to discontinue use of the affected chemicals and RMs.

## **10.0 Measurement Traceability**

### **10.1 Scope**

- Measurement traceability is required for net weights and purity determinations.

### **10.2 Net Weight**

#### **10.2.1 Balances**

The FC:

- Checks the performance of balances prior to being placed into service and at prescribed intervals thereafter, per 1-6.3.

#### **10.2.2 Reference Standards**

The FC:

- Uses reference standards (i.e., NIST-traceable weights) for balance performance verification. (See 1-6.3.1)

### **10.3 Purity**

#### **10.3.1 Instrumentation and Equipment**

The FC:

- Checks the performance of instrumentation and equipment prior to being placed into service and at prescribed intervals thereafter, per 1-6.

#### **10.3.2 Reference Materials**

The FC:

- Uses certified reference materials (CRMs) in analytical processes where uncertainty is estimated, per:
  - 1-7.0
  - 1-8.0
  - See SFL1 RM program SOP
- Obtains CRMs from sources determined to be reputable, based on factors including, but not limited to:
  - Demonstrated commitment to good laboratory practices

- Compliance with ISO/IEC 17025 or ISO 17034
- Past performance
- Proven technical competence in providing the chemical/physical characterization of RMs

## **10.4 Measurement Assurance**

### **10.4.1 Metrological Confirmation**

The FC:

- Performs balance calibration checks (1-6.3.1) and instrument performance verifications (1-6) to ensure the stability of laboratory equipment, and periodic drug RM verifications (1-8) to ensure the stability of RMs.

### **10.4.2 Statistical Control**

The FC:

- Monitors net weight measurement process variations through the monthly balance checks (1-6.3.1).
- Monitors the purity measurement process through the use of quality control samples (2-6).

### **10.4.3 Precision and Bias**

SFQ:

- Evaluates and incorporates the laboratory system's precision and bias into the uncertainty of measurement estimate for purity determinations.

## **10.5 Supplier Evaluation**

### **10.5.1 Analytical Supplies and Services**

The LD:

- Ensures analytical supplies and services are in accordance with LOM 75.
- Provides records of supplier evaluations and a list of approved suppliers to laboratory staff. (See Appendix 1H)

## 11.0 Reagent Reliability and Method Verification

The following policies and procedures have been established to ensure a consistent process for preparing, documenting, labeling, verifying, and disposing of reagents used in laboratory analyses.

### 11.1 Required References

#### 11.1.1 Tier 1 References

The FC:

- Prepares reagents according to the formulations in the following references:
  - *Analysis of Drugs Manual*, Appendix 1D – Color Test Reagent Preparation and Procedures
  - *Analysis of Drugs Manual*, Appendix 1E – Crystal and Precipitate Test Reagent Preparation and Procedures
  - *Analysis of Drugs Manual*, Appendix 1F – Thin Layer Chromatography
  - Microgram publications (DEA)
  - Laboratory Notes (DEA)

#### 11.1.2 Tier 2 References

The FC:

- Uses formulations found in the following references to prepare reagents if a reagent required for analysis is not listed in 1-11.1.1.
  - *Clarke's Analysis of Drugs and Poisons*, Edited by Moffat, Osselton, and Widdop
  - *Clarke's Isolation and Identification of Drugs*, E.G.C. Clarke
  - *Handbook of Chemical Microscopy*, Chamot and Mason
  - *Modern Microcrystal Tests for Drugs*, Fulton
  - *U.S. Pharmacopeia*
  - *Official Methods of Analysis*, AOAC
  - *Qualitative Inorganic Analysis*, Vogel
  - *Spot Tests in Organic Analysis*, Feigl
  - *Spot Tests in Inorganic Applications*, Feigl & Anger

- *Methods of Analysis*, IRS Publication
- *Clandestine Lab Basic Guide*, Clandestine Laboratory Investigating Chemists Association (CLIC)

### 11.1.3 Tier 3 References

The FC:

- Validates a reagent formula, according to 1-11.3 if a reagent preparation is not listed in 1-11.1.1 or 1-11.1.2, but is found in peer reviewed literature.

## 11.2 Developing Reagent Formulations

The LD:

- Requests approval for method development, in accordance with LOM 76.

The FC:

- Develops a new reagent, according to 1-11.3 if a reagent formula required for analysis is not available in the literature.

## 11.3 Validating Newly Developed Reagent Formulations

The FC:

- Tests at least five compounds and/or natural products commonly found with, or purported to be the compound of interest.
- Prepares a final report.
- Submits the final report through the laboratory management chain-of-command.

The LD:

- Submits the final report and associated data for approval in accordance with LOM 76.

## 11.4 Documenting Reagents

### 11.4.1 Single Use Reagents

The FC:

- Records reagent creation and verification in the “*Remarks*” finding for the appropriate LIMS test and includes:
  - Reagent name

- Lot number (commercial reagents only)
- Date created
- Verification results

#### **11.4.2 Multi-Use Reagents**

The FC:

- Records the following information in the LIMS test:
  - Reagent name
  - Lot number or laboratory traceable number

##### **11.4.2.1 Stock Containers**

The FC:

- Records the following information on the Reagent Reliability Verification Form on the SFDCC for each stock (primary) container.
  - Reagent name
  - Reference citation (See 1-11.1)
  - Laboratory traceable number
  - Prepared date
  - Preparer initials
  - Volume prepared
  - (Re)Verification date
  - (Re)Verification results
  - RM unique identifier used for (re)verification
  - Storage requirements (if applicable)
  - Disposition date

#### 11.4.2.2 Secondary Containers

The FC:

- Records the following information on the Reagent Reliability Verification Form - Secondary Containers on the SFDCC for each secondary container prepared from a verified stock container:
  - Reagent name
  - Laboratory traceable number
  - Date of transfer to secondary from stock
  - Analyst's initials
  - Volume transferred
  - (Re)Verification date
  - (Re)Verification results
  - RM unique identifier used for (re)verification
  - Disposition date

#### 11.4.2.3 Commercial (Purchased) Reagents

The FC:

- Records the following information on the Reagent Reliability Verification Form on the SFDCC once the manufacturer seals are broken on a commercial reagent.
  - Reagent name
  - Date opened
  - Analyst's initials
  - Manufacturer's reported reagent volume
  - Manufacturer name and lot number
  - (Re)Verification date
  - (Re)Verification results

NOTE: Instances where multi-container (i.e., 1 mL ampules) reagents are received from the same lot, verification will be performed on one container per lot number.



- RM unique identifier used for (re)verification
- Storage requirements (if applicable)
- Expiration date (if manufacturer provided)
- Disposition date

### 11.5 Labeling Containers

The FC:

- Labels reagent containers as follows:

	Reagent		Container	Labeling		
	Reagent Name	Analyst's Initials	Prepared Date	Transfer Date	Lab Traceable Number	Next Verification Date
Stock Containers	X	X	X	----	X	X
Secondary Containers	X	X	----	X	X	X
Commercially Prepared Containers	X	X	X	----	----	X

NOTE: The prepared date is the date the container is opened when the commercial reagent is used as is.

### 11.6 (Re)Verifying Reagents

The Reagent Monitor(s):

- Applies these requirements to any reagent in any container used for laboratory analysis.
- Verifies reagents are suitable for use immediately after preparing the reagent or after breaking the factory seal.
- Verifies reagents using RMs for all compounds expected to be identified using this reagent. Ensures that the results are consistent with the data found in approved reference literature or libraries to demonstrate the method's validity.
- Documents all RMs tested on the Reagent Reliability Verification Form in a reagent logbook.
- Re-verifies reagents within three months of the previous successful verification.
- Takes the following actions if a reagent does not produce expected results during (re)verification:

- Stock Containers:
  - Dispose of the stock reagent (See 1-11.8)
  - Dispose of secondary reagents traceable to the stock
  - Notify laboratory staff by e-mail of actions taken
  - Record the disposition date on the Reagent Reliability Verification Form
- Secondary Containers:
  - Dispose of the secondary reagent (See 1-11.8)
  - Record the disposition date on the Reagent Reliability Verification Form
  - Determine if the stock is affected and if so, take action as stated above for stock containers
  - Notify laboratory staff by e-mail of actions taken

The FC:

- Ensures all reagents used in casework were verified for the compound(s) being identified. Otherwise, analyzes a positive control concurrently.

### **11.7 Disposing of Reagents**

The Reagent Monitor(s):

- Disposes of a reagent as hazardous waste when it meets any of the following criteria: (See LOM 78)
  - Does not produce expected results during (re)verification
  - Drastically changes in appearance or composition
  - Is no longer needed

## **12.0 Peer Review of Forensic Chemist Examinations**

### **12.1 General Requirements**

- A minimum of three exhibits per FC are selected for peer review over the course of a fiscal year.

NOTE: The number of reviews selected for a FC not regularly performing examinations in the course of a fiscal year may be determined on a case-by-case basis.

- Peer review consists of technical and administrative review.
- FCs will not conduct peer reviews of their own work.

### **12.2 Conducting Peer Reviews**

The QAS or designee:

- Selects approved laboratory reports that have been reviewed within the past month.
- Assigns selected exhibit(s) to a FC for peer review.

The FC:

- Accesses the case file(s) in LIMS or retrieves the case file(s) of the specified exhibit(s).
- Conducts the peer review and documents findings using the LIMS Case File Peer Review Form or the SFL1 Case File Peer Review Form on the SFDCC.
- Provides the completed form to the QAS or designee for review.

### **12.3 Reviewing the Results of the Peer Review**

The QAS or designee:

- Reviews the results of the peer review for each exhibit.
  - If no corrections are required, notifies the FC and the SC that the review was completed.
  - If corrections are required, notifies the FC and SC of the necessary corrections.

The FC:

- Makes any corrections that are required as a result of the peer review.
- Creates an amended report (if needed) (see 2-11.8).
- Submits the corrections to the SC.

The SC:

- Reviews the FC's corrections.
- Approves the amended report (if created).
- Notifies the QAS once complete.

#### **12.4 Reporting the Results of the Peer Review**

The LQAM or designee:

- Compiles and maintains records to document which exhibits underwent peer review, including a summary of the findings and corrections made.
- Communicates the summary of findings to the entire chemist staff.
- Refers issues of concern (e.g., would have resulted in an analytical inconsistency or is a significant recurring finding) back to the Laboratory Quality Assurance Committee (LQAC) for further investigation. (See LOM 70 and 71)

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## 1.0 Evidence Analysis

### 1.1 Scope

- This chapter contains policy and procedures for the various aspects of evidence analysis.<sup>1</sup>
- Laboratory management must approve all deviations that do not fulfill these minimum requirements.

NOTE: Document approvals in the *Supervisory Approval* or *Deviations* test

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<sup>1</sup> Rodriguez-Cruz SE, Montreuil RS. Assessing the Quality and Reliability of the DEA Drug Identification Process. Forensic Chem 2017; 6: 36-43.

## **2.0 Determining Gross Weight and Opening Evidence**

### **2.1 Determining Gross Weight**

The Forensic Chemist (FC):

- Weighs the properly sealed evidence to determine the gross weight.

NOTE: Evidence packaged in accordance with the REDACTED (i.e., intact seals and complete labels) is considered properly sealed.

- Compares the obtained gross weight with the submitted gross weight. A witness is required if the weight differs by more than two grams or 0.2% of the gross weight of the evidence package, whichever is greater, from the reported weight of the submitting Special Agent (SA), Task Force Officer (TFO), or Diversion Investigator (DI), or if there is no gross weight recorded either on the evidence package or on the DEA-7.
  - A Supervisory Chemist (SC) or another FC witnesses the evidence gross weight, prior to breaking the seal by entering one's username and password in the *Gross Weight* test.
  - Take appropriate follow-up action, which may include referral to the Office of Professional Responsibility (OPR).
- Reports the gross weight in LIMS and on the DEA-113.

### **2.2 Opening Evidence**

#### **2.2.1 Properly Sealed Evidence**

The FC:

- Opens plastic sealed evidence envelopes (PSEE) and manila envelopes by cutting along the edge opposite the SA's, TFO's, or DI's evidence seal, creating a separate strip.
  - Annotate FC's initials, date opened, and a unique identifier on the strip.
  - Place the annotated strip inside the original evidence envelope.
  - Annotate the PSEE label with the date opened and any other applicable information.
  - Record the original condition of the seal and the date opened in the *Description of Evidence* test.
- Opens boxes and cans by breaking the SA's, TFO's, or DI's affixed evidence seal(s).
  - Annotate the affixed evidence label with the date opened and any other applicable information.

- Record the original condition of the seal(s) and the date opened in the *Description of Evidence* test.

### 2.2.2 Improperly Sealed Evidence

The FC:

- Notifies a manager when evidence is not packaged in accordance with REDACTED (i.e., the SA's seals are not intact).

The SC:

- Witnesses the condition of the evidence by entering one's username and password in the *Description of Evidence* test.
- Decides if the evidence will be returned to the vault or be analyzed.

### 2.3 Describing the Evidence

The FC:

- Compares the physical evidence with the description reported by the submitting Investigating Agency (IA) and describes the evidence as received from the outermost packaging to the innermost contents.

NOTE: Descriptions that are too long to fit in the LIMS field may be added as an attachment to the test (as a PDF or an image file).

- Selects "Yes" in the *Consistent with Paperwork?* finding in the *Description of Evidence* test when the physical evidence is consistent with the description.
- Selects "No" in the *Consistent with Paperwork?* finding in the *Description of Evidence* test when the physical evidence differs significantly from the description.

- Obtain a witness to the description discrepancy.

NOTE: The witness verifies the FC's description in the *Description of Evidence* test by entering one's username and password.

- Discuss the discrepancy with an SC and contact the SA, TFO, or DI, if necessary, in an attempt to resolve any significant differences.
- Document the results of the contact in LIMS.
- Updates the *Lab Exhibit Description* in the *Organize My Work* pane to include a brief description of the innermost packaging and contents. If all of the packages will not be opened, the FC provides a general description of the exhibit.

## 2.4 Handling Interior Packaging

The FC:

- Marks interior packages with initials, date, and unique identifier. Alternatively, places the interior package(s) into a substitute container marked with initials, date, and unique identifier.
  - If latent print examination is requested, place interior packaging into a substitute container that has been marked with initials, date, and unique identifier. Use caution to avoid obliteration of any latent prints that might be present.

NOTE: For bulk exhibits, it is only necessary to mark the threshold portion.

## 2.5 Photographing Evidence

The FC:

- Uses a digital camera or digital video camera.
- Photographs and/or records item(s) seized as evidence or essential area(s) where evidence was obtained, as appropriate, during field operations (i.e., clandestine laboratories, IONSCANS, vacuum searches).
- Photographs evidence during analysis in the laboratory when required, when necessary to document any unusual physical feature(s) prior to processing, or for *REDACTED*.
- Attaches all photographs *REDACTED* to the *Image* finding in the *Description of Exhibit and Sampling* test.
- Includes a self-documenting sign in all photographs *REDACTED* which contains the following:
  - Unique identifier
  - Date photographs are taken
  - Location of the seizure (bulk evidence only)
  - Laboratory
  - Handwritten initials of photographer
- Positions the sign so that it appears in all the photographs.
- Positions an object used to measure the physical size of the seizure, such as a ruler or yardstick, in all photographs.
- Assembles or stacks the evidence, when appropriate, so that it makes a clear, visual display of the individual units.

- Photographs the entire display, and, if the evidence is in closed containers (e.g. evidence boxes), several open containers to display the contents.

## **2.6 Bulk Evidence**

The FC:

- Weighs and photographs evidence suspected as having a net weight exceeding the threshold amount, as follows:
  - Photograph and/or record the entire seizure (see 2-2.5).
  - Determine the gross weight of the total exhibit.

## **2.7 Creating Sub-Exhibits (Splits)**

The FC:

- Creates a new sub-exhibit(s) through *Organize My Work, Add Lab Exhibit*.
- Enters the *Lab Exhibit* as “*Exhibit Number.OX*” (e.g., 1.01, 1.02, etc.) in the *Lab Exhibit Details* section.
- Describes the sub-exhibit’s innermost packaging (if applicable) and appearance in the *Description*.
- Selects the radial button for *Place in Current Container*.
- Changes the original *Lab Exhibit Number* and *Lab Exhibit* description to reflect the sub-exhibit number and the sub-exhibit description.

### 3.0 Determining Net Weight and Uncertainty of Measurement Estimates

The FC:

- Determines the net weight, volume, or unit count by direct measurement of all units in the exhibit.

OR

- Obtains the total net weight, volume, or unit count by extrapolation, when direct measurement of all units is not practical and provided that some or all units in the exhibit contain uniform amounts of material or uniform packaging.

OR

- Divides the exhibit into sub-groups, when the units in an exhibit are not uniform, but can be divided into sub-groups of uniform size or packaging (e.g., an exhibit containing 150 10 mL vials and 50 20 mL vials).
- Obtains the net weight, volume, or unit count for each sub-group. The total net weight, volume, or unit count for the exhibit is the sum of all the sub-group measurements.

#### 3.1 Observing Minimum Weight Thresholds

The FC:

- Ensures that established minimum weight thresholds are observed when performing net weight measurements. (See Appendix 2F)

#### 3.2 Calculating Net Weight, Solid Dosage Count, and Volume

##### 3.2.1 Powders (mixtures of powders and materials), gummy samples, and plant material

###### 3.2.1.1 One to nine units per exhibit or sub-group

The FC:

- Determines the total net weight by direct measurement of the contents of all units.

###### 3.2.1.2 Ten or more units per exhibit or sub-group

The FC:

- Determines the total net weight by direct measurement of the contents of all units.

OR

- Determines the total net weight by **contents** extrapolation as follows:

- Individually weigh nine randomly selected units. (See 2-3.5, Policy exception #2)
- Calculate the average net weight per unit.
- Obtain the total net weight by multiplying the average weight per unit by the total number of units.

OR

- Determines the total weight by **container** extrapolation as follows:
  - Determine the total gross weight of the units by direct measurement of all units.
  - Individually weigh nine randomly selected empty containers. (See 2-3.5, Policy exception #2)
  - Calculate the average weight per empty container.
  - Obtain the total weight of the empty containers by multiplying the average weight per container by the total number of units in the exhibit.
  - Obtain the total net weight by subtracting the total weight of the empty containers from the total gross weight for all units.

### 3.2.2 Dosage Units

NOTE 1: For capsule exhibits, the net weight does not include the capsule shell.

NOTE 2: For impregnated paper, the net weight is to include the paper.

#### 3.2.2.1 One to nine units per exhibit or sub-group

The FC:

- Counts and weighs all units directly.

#### 3.2.2.2 Ten or more units per exhibit or sub-group

The FC:

- Counts and weighs all units directly.

OR

- Weighs all units directly; and
- Determines the total unit count by extrapolation, as follows:
  - Individually weigh nine randomly selected dosage units.



- Calculate the average weight per dosage unit.
- Obtain the total number of dosage units in the exhibit by dividing the total net weight by the average weight per dosage unit.

OR

- For capsules, determines the total weight by **container** or **contents** extrapolation. (See 2-3.2.1.2)

### 3.2.3 Liquids

#### 3.2.3.1 One to nine units per exhibit or sub-group

The FC:

- Determines the total net weight by direct measurement of the contents of all units.
- Determines the density of the liquid by accurately weighing a minimum of 1.0 mL of the composite, using Class-A volumetric glassware and an analytical balance.
- Calculates the total net volume using the total net weight and the composite's density.

#### 3.2.3.2 Ten or more units per exhibit or sub-group

The FC:

- Determines the total net weight by direct measurement of the contents of all units.
- Determines the density of the liquid by accurately weighing a minimum of 1.0 mL of the composite, using Class-A volumetric glassware and an analytical balance.
- Calculates the total net volume using the total net weight and the composite's density.

OR

- Determines the total net weight and volume by **container** extrapolation. (See 2-3.2.1.2)
- Determines the density of the liquid by accurately weighing a minimum of 1.0 mL of the composite, using Class-A volumetric glassware and an analytical balance.
- Calculates the total net volume using the total net weight and the composite's density.

### 3.2.4 Internal Body Carry Exhibits

The FC:

- Determines the total net weight of the exhibit by direct measurement of the contents of all units.

OR

- Determines the total net weight by **contents** extrapolation. (See 2-3.2.1.2)

NOTE: With supervisory approval, fewer units may be weighed to limit exposure.

### 3.3 Uncertainty of Measurement Estimate Determination

The FC:

- Reviews the Uncertainty Calculator and applicable Worksheet in LIMS to ensure the values from each weighing event are entered correctly.

NOTE: Some circumstances may require use of the Legacy Calculator.

- Accepts the calculated uncertainty when:
  - For extrapolation cases, the relative standard deviation (RSD) obtained from the nine individual weights measured is 10% or less.
  - The calculated expanded relative uncertainty (U/NW) associated with the total net weight is 25% or less.
- Pursues alternative approaches to net weight determination (e.g., use of a higher precision balance, extrapolation by container instead of contents, weighing units by groups of higher uniformity, etc.) when the acceptance criteria are not met.

### 3.4 Reporting Net Weight and Uncertainty Estimates

The FC:

- Reports all final net weight and uncertainty results in LIMS and on the DEA-113.
- Reports the final expanded uncertainty values after rounding to one significant figure using ISO/NIST rounding rules:
  - When the digit following the one to be retained is less than five, keep the retained figure unchanged. Example: To one significant figure, 2.441 becomes 2.
  - When the digit following the one to be retained is greater than five, increase the retained figure by one. Example: To one significant figure, 0.267 becomes 0.3.
  - When the digit following the one to be retained is five and at least one of the following digits is greater than 0, increase the retained figure by one. Example: To one significant figure, 0.4507 becomes 0.5
  - When the digit following the one to be retained is five and all of the following digits are 0, keep the retained figure unchanged if it is even or increase by one if it is odd. Examples: To one significant figure, 3.500 becomes 4, and 4.500 becomes 4 (the final digit is always even).

- Reports the final net weight to the same precision as the final expanded uncertainty (same number of decimal places or same level of significance).
- Includes a statement, selected from Appendix 2B, that describes the procedure used for net weight determination and the associated uncertainty, if determined, in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113.

NOTE: The coverage factor used in calculating all uncertainty values corresponds to a 95% level of confidence. Do *not* report the coverage factor (*k* value) on the DEA-113; it is available in the case file documentation.

- Reports total net solid dosage unit counts and total net volumes in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113.

NOTE: Do not include measurement uncertainty values associated with these quantities on the DEA-113.

### 3.5 Exceptions

- Policy exception #1: For submissions representing a part of a larger seizure REDACTED net weight uncertainty estimates are not required to be calculated or reported.
- Policy exception #2: When extrapolating and combining the net weights of *two* or *more* sub-groups containing 10 or more units each, it is acceptable to individually weigh less than nine units per sub-group to avoid opening more units than those to be opened for analysis.
- Policy exception #3: Since uncertainty is not required for exemplar exhibits, for 1B-K samples submitted in similar packaging, the net weight may be obtained by weighing all samples full, and subtracting the extrapolated weight of one empty container.

### 3.6 Revising UMEs

The Office of Forensic Sciences Quality Assurance Section (SFQ):

- Reviews and updates the mass uncertainty values associated with each balance type every accreditation cycle using performance verification data from all DEA laboratories.

## 4.0 The Evidence Sampling Plan

REDACTED

The Evidence Sampling Plan (ESP) for Qualitative Analysis:

- Includes procedures and requirements for FCs to report the identification of analyte(s) in a drug exhibit.
- Requires a non-statistical sampling approach for submissions having fewer than 10 units.
- Requires a statistical sampling approach for submissions consisting of 10 or more units (non-exemplar). This approach:
  - Requires random sampling.
  - Is based on the probability theory of the hypergeometric distribution.
  - Allows a consistent mathematical foundation for conclusions concerning a high proportion of the exhibit's population.
  - Allows an inference that a high proportion of units in an exhibit's population contain the target analyte(s) at a minimum 95% level of confidence.
- Includes procedures for sampling residue exhibits.
- Includes procedures for arbitrary sampling when no inference will be made on the population.

The ESP for Composite Formation and Quantitative Analysis:

- Defines procedures and requirements for FCs to form composites, including combination of all units and incremental sampling. The incremental sampling approach:
  - Requires random sampling. (See Appendix 2A)
  - Is based on the 2014 European Network of Forensic Science Institutes – Drug Working Group (ENSFI-DWG) Guidelines.
  - Allows a population inference on the purity of the analyte(s).

The FC:

- Separates the exhibit into sub-exhibits as needed.

NOTE: Separation may be based on different colors, markings, expected target analyte(s), bi-layer liquids, upon the results of chemical testing, etc.

- Follows the ESP for all exhibits or sub-exhibits.

- Documents the reason and obtains approval in advance for any deviations from the ESP, according to Appendix 2F.

The SC:

- Reviews deviation request.
- Approves or denies deviation from the ESP.

#### **4.1 Sampling for Qualitative Testing (Identification)**

##### **4.1.1 Exhibits Containing 1-9 Units**

The FC:

- Selects all units.
  - For single unit exhibits, proceed directly to composite formation according to 2-4.3.
  - For solid dosage units (except capsules), photograph the front and back of an intact unit prior to analysis.
- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met (2-4.2).
- Forms a composite according to 2-4.3.

##### **4.1.2 Exhibits Containing 10 or More Units**

###### **4.1.2.1 Powders, crystalline materials, liquids and solutions, gummy substances, and body carries**

The FC:

- Uses Table 1 to determine the number of units to be randomly selected.
- Segregates or labels (e.g., numbers) each unit selected.
- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met. (See 2-4.2)
- Forms a composite according to 2-4.3.

###### **4.1.2.2 Dosage Units**

NOTE: Impregnated paper dosage unit size is defined as a ¼" x ¼" square unless otherwise perforated or marked (e.g., a drawn grid or repeated design).

#### 4.1.2.2.1 Single Container

The FC:

- Uses Table 1 to determine the number of units to be randomly selected.
- Segregates or labels (e.g., numbers) each unit selected.
- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met (2-4.2).
- Forms a composite according to 2-4.3.

#### 4.1.2.2.2 Multiple Containers

The FC:

- Selects the sample size as follows:
  - Use Table 1 to determine the number of units to be randomly selected based on the total number of units in the exhibit.
  - Sample from as many containers as possible.

Example 1: For an exhibit containing a total of 1000 dosage units in 12 containers, remove two units from each container and one additional unit from each of four randomly selected containers, for a total of 28 units.

Example 2: For an exhibit containing a total of 6500 dosage units in 50 containers, remove one unit from each of 29 randomly selected containers.

- Segregates or labels (e.g., numbers) each unit selected.
- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met. (See 2-4.2)
- Forms a composite according to 2-4.3.

#### 4.1.2.3 Plant Materials

##### 4.1.2.3.1 Exemplar Exhibits

The FC:

- Selects all units.

NOTE: 2 kg samplings of exhibits of synthetic drugs on plant material are sampled as non-exemplar exhibits. (See 2-4.1.2.3.2)

- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met. (See 2-4.2)

#### 4.1.2.3.2 Non-exemplar Exhibits

The FC:

- Uses Table 1 to determine the number of units to be randomly selected.
- Segregates or labels (e.g., numbers) each unit selected.
- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met. (See 2-4.2)

#### 4.1.3 Other Exemplar Exhibits (non-Plant Material)

The FC:

- Selects all units.

NOTE: For 2 kg samplings of exhibits of synthetic drugs use Table 1 to determine the number of units to be randomly selected.

- Analyzes each selected unit as directed in 2-5.
- Evaluates results to determine if the objective of the ESP has been met. (See 2-4.2)
- Forms a composite according to 2-4.3.

#### 4.1.4 Residue Exhibits

The FC:

- Treats commingled items submitted in the same evidence container as one unit.

NOTE: Items of evidence that are closed containers (e.g., plastic bags with residue) may be treated as commingled.

- Sub-exhibits physically segregated items (i.e., evidence items purposefully placed in secondary containers, etc.).
- Selects at least one item from the exhibit or each sub-exhibit for testing.
- Analyzes the selected item(s) according to 2-5.

#### 4.1.5 Arbitrary Sampling

The SC:

- Obtains written concurrence from the customer.
- Documents communication in LIMS.
- Documents approval of the deviation from the ESP in the *Description of Exhibit and Sampling* test in LIMS.

The FC:

- Obtains supervisory approval.
- Selects at least one unit and designates it as sub-exhibit X.01.
- Separates remaining units as sub-exhibit X.02 for no analysis.
- Analyzes the selected unit(s) as directed in 2-5.
- Forms a composite according to 2-4.3.
- Refers to Appendix 2B (Scenario G) for sampling statement.

#### 4.2 Meeting the Objective of the Sampling Plan

##### 4.2.1 Positive Results for All Units

The FC:

- Concludes that the objective of the ESP has been met when all selected units have tested positive for the same target analyte(s).
  - The same controlled substance(s) is identified in each of the individual units selected for testing.

NOTE: For exhibits containing 10 or more units, when additional controlled substances are confirmed in some but not all units selected for testing, the population inference, 90% of the population at the 95% level of confidence, cannot be made for these additional substances. Refer to Appendix 2B (Scenario C) for the sampling statement.

- In the absence of controlled substances, the same listed chemical(s) OR non-controlled substance(s) is identified in each of the individual units selected for testing.
  - Refer to Appendix 2B (Scenarios A and B) for sampling statements.
- Forms a composite according to 2-4.3.



#### 4.2.2 Negative Result Observed in One Unit

The FC:

- Concludes that the objective of the ESP has not been met when a negative result is observed for one of the selected units.

NOTE: A result is considered negative when a unit does not contain the target analyte(s) confirmed in the rest of the units tested.

- Proceeds according to one of the following based on the number of units in the original exhibit:
  - 2-9: Separate the exhibit into sub-exhibits.
  - 10-59:
    - Analyze all units and separate the exhibit into sub-exhibits.
    - OR
    - Perform arbitrary sampling according to 2-4.1.5 and create one or more sub-exhibits.
  - 60 or more:
    - Use one of the two options listed for 10-59 units.
    - OR
    - Report qualitative result(s) as obtained without testing additional units. Refer to Appendix 2B (Scenario D) for the sampling statement.
- Forms a composite according to 2-4.3.
- Documents course of action in the *Description of Exhibit and Sampling* test in LIMS.

#### 4.2.3 Negative Results Observed in Two or More Units

The FC:

- Concludes that the objective of the ESP has not been met when a negative result is observed for two or more of the selected units.

NOTE: A result is considered negative when a unit does not contain the target analyte(s) confirmed in the rest of the units tested.

- Proceeds according to one of the following based on the number of units in the original exhibit:
  - 3-9: Separate the exhibit into sub-exhibits.

- 10 or more:

- Analyze all units and separate the exhibit into sub-exhibits.

OR

- Perform arbitrary sampling according to 2-4.1.5 and create one or more sub-exhibits.

OR

- Report qualitative result(s) as obtained without testing additional units. No population inference is made. Refer to Appendix 2B (Scenario E) for a sampling statement based on the final number of positive results confirmed.

- Forms a composite according to 2-4.3.
- Documents course of action in the *Description of Exhibit and Sampling* test in LIMS.

#### **4.2.4 No Analytes Observed for All Units**

The FC:

- Concludes that the objective of the ESP has been met when no reportable analyte(s) are identified in any of the selected units
- Does not form a composite. (See 2-4.3) Refer to Appendix 2B (Scenario F) for sampling statement.

#### **4.3 Sampling to Form Composites**

The FC:

- Forms a composite according to Appendix 2C for all exhibits (or sub-exhibits) except for the following:
  - Exhibits that are not amenable to mixing or grinding
  - Residues
  - Sub-lingual films, blotter paper, patches, etc.
  - Plant materials
  - Substances applied to plant materials
  - No analyte(s) observed for all units (See 2-4.2.4)

NOTE: Refer to 2-6.5 for preparation of solutions for the quantitation of exhibits where no composite is required.

- Analyzes the composite per 2-5.

## 5.0 Qualitative Analysis

### 5.1 General Analysis Requirements

The FC:

- Uses validated qualitative methods during casework analysis.
- Evaluates the data of the unknown for suitability in meeting acceptance criteria in 2-5.10 prior to comparison to the known.
- Bases all conclusions on reviewable data which support the reported identifications. Examples of reviewable data include spectra, chromatograms, photographs, or detailed annotations for color, precipitate, and microscopic tests.
  - Any data or observation which is inconsistent with the identification must be fully explained, or the compound cannot be reported.
- Compares the sample results to data from positive controls (i.e., DEA laboratory reference materials) analyzed under the following conditions:
  - For color and precipitate tests: Within three months or concurrently. (See 1-11)
  - For thin-layer chromatography (TLC): Concurrently. (See 2-5.10.3)
  - For gas chromatography (GC), liquid chromatography (LC), and capillary electrophoresis (CE) tests: Within a month using the same method and instrument.
    - Positive control data must meet the acceptance criteria found in 1-3.1.1.2.
  - For mass spectrometry (MS), infrared (IR) spectroscopy, Raman spectroscopy, and nuclear magnetic resonance (NMR) spectroscopy: Using the same method and instrument.
    - Positive control data must meet the acceptance criteria found in 1-3.2.1.2.

NOTE: Any timeframes listed above may be further limited by the method validation.

- Includes the following in positive control instrumental data: instrument identifier, reference material unique identifier, and date and method of analysis.
- For structural elucidation requests, contacts SFL1.
- Bases identifications on established general acceptance criteria per 2-5.10.
- Uses negative controls (blanks) with instrumental and chemical tests to verify that the solvents, reagents, and instruments are free of contamination.
- Analyzes a negative control immediately prior or concurrently (TLC and color tests) to the sample.

NOTE 1: Blanks are not needed between units when analyzing multi-unit submissions with the same exhibit number (including A-K submissions). If sub-exhibiting occurs as a result of the pre-composite analysis, retesting with additional negative controls is not needed.

NOTE 2: For NMR, blanks are not needed between exhibits. However, a single blank must be associated with each batch of samples using the same deuterated solvent source and analyzed on the same day.

NOTE 3: See 2-5.10 for specific negative control requirements for individual techniques.

## **5.2 Minimum Analysis Requirements**

### **5.2.1 Single Unit Analysis**

The FC:

- Follows the general requirements in 2-5.1.
- Forms a composite per 2-4 and analyzes at least two samplings.

NOTE: Samplings cannot be analyzed on the exact same instrument.

- Identifies analyte(s) using at least two different and independent tests, incorporating at least one confirmatory technique.

NOTE: Hyphenated techniques may be considered independent tests provided that the results from each are used. However, the use of a hyphenated technique will not satisfy the requirement for analysis of two different samplings.

- Analyzes the composite using a separation technique.

NOTE 1: The results from a separation technique are used to assess the presence of multiple components.

NOTE 2: The results may be used (but are not required) as one of the two tests needed for identification of individual analytes. If used, results must fulfill acceptance criteria for the separation technique.

- Reports “No Controlled Substances” as the final analytical result when no reportable analytes are identified or when the analytical data is insufficient for confirmation of any compound.

### **5.2.2 Multiple Units: Pre-Composite Analysis**

The FC:

- Follows the general requirements in 2-5.1.
- Analyzes at least two samplings from each selected unit.

NOTE: Samplings cannot be analyzed on the exact same instrument.

- Identifies the target analyte(s) in each unit using at least two different and independent tests, incorporating at least one confirmatory technique.

NOTE 1: Non-controlled substances may be identified during the pre-composite analyses if requirements in 2-5.5 are met.

NOTE 2: Hyphenated techniques may be considered independent tests provided that the results from each are used. However, the use of a hyphenated technique will not satisfy the requirement for analysis of two different samplings.

- Analyzes each unit using a separation technique.

NOTE 1: The results from a separation technique are used to assess the presence of multiple components.

NOTE 2: The results may be used (but are not required) as one of the two tests needed for identification of individual analytes. If used, results must fulfill acceptance criteria for the separation technique.

- Reports “No Controlled Substances” as the final analytical result when no reportable analytes are identified or when the analytical data is insufficient for confirmation of any compound.

### 5.2.3 Multiple Units: Composite Analysis

The FC:

- Follows the general requirements in 2-5.1.
- Tests at least one sampling. The results may be used for any of the following purposes:
  - Identifying additional controlled substance(s)
  - Identifying non-controlled substances or supplementing the partial identification of non-controlled substances during pre-composite testing
  - Evaluating the sample for adulterants at or above 1% level
  - Determining salt form of a target analyte
  - Quantifying a target analyte
  - Determining the optical isomer of a target analyte

NOTE: For positional isomers, geometric isomers, and diastereomers, see 2-5.4.

### 5.3 Identifying Controlled Substances

The FC:

- Identifies controlled substances present in a sample, provided that a sufficient amount of material exists and all requirements have been met.
  - If a controlled substance appears to be present as the result of naturally occurring alkaloids, incomplete reactions or sample breakdown, the substance is not identified or reported unless it is the predominant controlled substance in the exhibit.
  - The general analysis requirements and criteria for identifying controlled substances also apply to the identification of potential controlled substance analogues.

- Makes identifications based on all of the following minimum criteria:

- The results from testing at least two samplings are used for identification.
- Two orthogonal techniques are used, incorporating at least one confirmatory technique.

NOTE 1: Hyphenated techniques may be considered independent tests provided that the results from each are used. However, the use of a hyphenated technique will not satisfy the requirement for analysis of two different samplings.

NOTE 2: For newly encountered substances, potential limitations should be considered when selecting an analytical scheme.

- Results must be from either pre-composite or composite testing, not from a combination of both.
- Determines salt form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., cocaine and methamphetamine), unless impractical to do so.
  - Salt form determination may be performed on the composite.
  - All salt forms determined are reported.

### 5.4 Determining Isomers

The FC:

- When determining positional, geometric, or diastereomeric isomers, identifies and determines the isomer on each unit, provided sufficient amount of material exists.
  - If sufficient material does not exist, identifies the compound, including determination of positional, geometric, and diastereomeric designation, in the composite.
  - Identification and determination of the isomer must be from either pre-composite or composite testing, not from a combination of both.

- Determines optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., methamphetamine), unless impractical to do so.
  - Performs optical isomer determination on the composite.

NOTE: If the total methamphetamine hydrochloride concentration is greater than or equal to 80%, isomer determination is required. Methamphetamine optical isomers are reported according to procedures in Appendix 2D.

### 5.5 Identifying Adulterants

The FC:

- Identifies adulterants, if present at a level of 1% or greater.
- Makes identifications based on all of the following minimum criteria:
  - The result from testing at least one sampling is used for identification.
  - Two orthogonal techniques are used, incorporating at least one confirmatory technique.

NOTE: Hyphenated techniques may be considered independent tests provided that the results from each are used.

- Results may be from pre-composite testing, composite testing, or a combination of both.

### 5.6 Identifying Diluents

The FC:

- Identifies diluents when requested by the customer and approved by laboratory management.
- When needed, makes identifications based on all of the following minimum criteria:
  - The result from testing at least one sampling is used for identification.
  - Either one confirmatory technique or two presumptive techniques are used.

### 5.7 Analyzing Residue Exhibits

The FC:

- Follows the general requirements in 2-5.1.
- Analyzes the exhibit as directed in 2-5.2.1 or 2-5.2.2, if there is sufficient sample for two independent samplings.

OR



- Makes identifications based on all of the following minimum criteria:
  - The result from testing one sampling (e.g., rinse, swab) using a procedural blank.
  - Two different and independent techniques are used to test the single sampling, incorporating at least one confirmatory technique. For example, the single sampling could be analyzed using GC-MS provided the results from both the GC and MS portions are used.
- Segregates and retains the procedural blank and sample vials as reserve evidence when there is insufficient material for two samplings.

## **5.8 Identifying Marijuana, Hashish, and Marijuana Seeds**

For the analysis of suspected cannabis plant material exhibits seized on or after December 20, 2018, follow SOP-CH-001.

The FC:

- Follows the general requirements in 2-5.1.
- Gross forms not described below (e.g., liquid, oil, wax, powder, edibles, etc.) are analyzed per 2-5.2 through 2-5.6.

### **5.8.1 Marijuana**

The FC:

- Identifies marijuana based on all of the following minimum criteria:
  - A macroscopical examination to observe that the gross form of the substance is plant material.
  - A microscopical examination to observe cystolithic hairs.  
  
NOTE: Effervescence of the calcium carbonate crystal (i.e., cystolith at the base of the hair) in dilute acid may be performed.
  - A Duquenois-Levine color test where the purple/violet/blue color is extracted into the chloroform layer.
  - A chromatographic test (e.g., TLC, GC, etc.) or mass spectrometry test supporting the presence of at least one of the following: cannabinol, tetrahydrocannabinol (THC), or cannabidiol.
- Reports “Marijuana” on the DEA-113.

### 5.8.2 Cannabis Resin (Hashish)

The FC:

- Identifies hashish based on all of the following minimum criteria:
  - A macroscopical examination to observe that the gross form of the substance is gum-like or resinous, brown material.
  - A microscopical examination to observe cystolithic hairs as described in 2-5.8.1.
  - A Duquenois-Levine color test where the purple/violet/blue color is extracted into the chloroform layer.
  - A mass spectrometry test supporting the presence of one or more of the tetrahydrocannabinols (THC), and at least two of the following: cannabinol, cannabidiol, cannabigerol, cannabicyclol, or cannabichromene.
- Reports “Hashish” on the DEA-113.

### 5.8.3 Cannabis Seed (Marijuana Seeds)

The SC:

- Approves germination of marijuana seeds when demonstration of the viability is needed.

The FC:

- Follows the procedures outlined in Appendix 2E.
- Adds a statement to the Remarks of the *Observations, Results, and Conclusions* section of the DEA-113 to reflect the following:
  - The total number of seeds (actual or extrapolated count)
  - The number of seeds selected for germination
  - The number of viable seeds
  - The number of plants grown and identified as marijuana

Example: Exhibit 1 contains 250 seeds. Selected 29 seeds for germination and 18 were viable; 15 of 18 viable seeds produced plants that were individually identified as marijuana.

- Reports “Marijuana Seeds” on the DEA-113.

## 5.9 Identifying Opium

The FC:

- Identifies opium based on all of the following minimum criteria:
  - A macroscopical examination to observe that the gross form of the substance is a gum-like or resinous, brown material.
  - The results from testing at least two samplings are used for identification.  
  
NOTE: Samplings cannot be analyzed on the exact same instrument.
  - Two different and independent tests are used, incorporating at least one confirmatory technique.  
  
NOTE: Hyphenated techniques may be considered independent tests provided that the results from each are used. However, the use of a hyphenated technique will not satisfy the requirement for analysis of two different samplings.
  - At least four of the following are confirmed: codeine, morphine, thebaine, papaverine, or noscapine.
- Reports “Opium” on the DEA-113.
  - Also reports any adulterants identified per 2-5.5.

## 5.10 Use of Qualitative Tests and Techniques and Acceptance Criteria

- Portable instrumentation is intended for field use only and is not to be used for casework.

The FC:

- Uses the qualitative tests and techniques described in this section to identify controlled and non-controlled substances.
- Bases identifications on the general acceptance criteria for each test.
- Uses spectral processing tools, as needed.
  - Scale normalization may be applied for comparison of sample spectra and positive controls.
  - Background subtraction may be applied for comparison of sample spectra and positive controls.
  - Spectral subtraction may be used to eliminate the influence of interfering or co-eluting substances.

- Uses laboratory-specific protocols approved per 1-6 (performance verification) for any qualitative test not included in this section.

#### **5.10.1 Color Tests**

The FC:

- Uses color tests as presumptive techniques in the qualitative analysis of controlled and non-controlled substances.
- Follows procedures in Appendix 1D: Color Test Reagent Preparation and Procedures.
- Ensures all reagents used in casework were verified for the compound(s) being identified. Otherwise, concurrently analyzes a positive control.
- Accepts a result when the color change observed for the sample is consistent with the positive control.

#### **5.10.2 Precipitate Tests**

The FC:

- Uses precipitate tests as presumptive techniques in the qualitative analysis of controlled and non-controlled substances.
- Follows procedures in Appendix 1E: Crystal and Precipitate Test Reagent Preparation and Procedures.
- Ensures all reagents used in casework were verified for the compound(s) being identified. Otherwise, analyzes a positive control concurrently.
- Accepts a result when the precipitate observed for the sample is consistent with the positive control.

#### **5.10.3 Thin Layer Chromatography**

The FC:

- Uses TLC as a separation technique in the qualitative analysis of mixtures.
- Follows procedures in Appendix 1F: Thin Layer Chromatography.
- Uses TLC as a presumptive test for identification purposes by comparing the retention factor of the analyte to that of a positive control that has been concurrently analyzed.
- Accepts a result when:
  - The retention factor of the analyte is within 5% of the positive control.

- The spot color of the analyte is consistent with the positive control.

#### 5.10.4 Gas Chromatography

The FC:

- Uses GC as a separation technique in the qualitative analysis of mixtures.

NOTE: GC as a separation technique may be used for the isomeric determination of compounds.

- Uses GC as a presumptive test for identification purposes by comparing the retention time (or relative retention time) of the analyte to that of a positive control by either direct comparison or by co-analysis of the positive control and sample.

NOTE: Retention times are measured by integration of the peak.

- For isomer (optical, positional, geometric, and diastereomer) determinations, co-analysis is required when more than one peak is within the acceptance criteria window for direct comparison. Co-analysis is not required when more than one peak is within the acceptance criteria window for direct comparison and a confirmatory test is used to distinguish between isomers.
- Accepts a result when a single peak with a clear, non-splitting apex is observed.
- Accepts a result when the peak-to-peak signal-to-noise ( $S/N_{pk-pk}$ ) is greater than 3.
- Accepts a result for direct comparison when the retention time of the positive control and sample are within 0.1 minutes. If using relative retention time, the acceptance criterion is 1%.
- Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

#### 5.10.5 Liquid Chromatography

The FC:

- Uses LC as a separation technique in the qualitative analysis of mixtures.

NOTE: LC as a separation technique may be used for the isomeric determination of compounds.

- Uses LC as a presumptive test for identification purposes by comparing the retention time (or relative retention time) of the analyte to that of a positive control by either direct comparison or by co-analysis of the positive control and sample.

NOTE: Retention times are measured by integration of the peak.

- For isomer (optical, positional, geometric, and diastereomer) determinations, co-analysis is required when more than one peak is within the acceptance criteria window for direct comparison. Co-analysis is not required when more than one peak is within the acceptance

criteria window for direct comparison and a confirmatory test is used to distinguish between isomers.

- Accepts a result when a single peak with a clear, non-splitting apex is observed.
- Accepts a result when the  $S/N_{pk-pk}$  is greater than 3.
- Accepts a result for direct comparison when the retention time of the positive control and sample are within 0.1 minutes for LC or 0.3 minutes for LC-MS. If using relative retention time, the acceptance criterion is 1% for both LC and LC-MS.
- Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

#### 5.10.6 Capillary Electrophoresis

The FC:

- Uses CE as a separation technique in the qualitative analysis of mixtures.

NOTE: CE as a separation technique may be used for the isomeric determination of compounds.

- Uses CE as a presumptive test for identification purposes by comparing the migration time (or relative migration time) of the analyte to that of a positive control by either direct comparison or by co-analysis of the positive control and sample.

NOTE: Migration times are measured by integration of the peak.

- For isomer (optical, positional, geometric, and diastereomer) determinations, co-analysis is required when more than one peak is within the acceptance criteria window for direct comparison. Co-analysis is not required when more than one peak is within the acceptance criteria window for direct comparison and a confirmatory test is used to distinguish between isomers.
- Accepts a result when a single peak with a clear, non-splitting apex is observed.
- Accepts a result when the  $S/N_{pk-pk}$  is greater than 3.
- Accepts a result for direct comparison when the migration time of the positive control and sample is within 0.3 minutes. If using relative migration time, the acceptance criterion is 1%.
- Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

#### 5.10.7 Infrared Spectroscopy

The FC:

- Uses IR as a confirmatory technique in the qualitative analysis of controlled and non-controlled substances.

- Uses IR as a confirmatory test for identification purposes by comparing the spectrum of the sample to that of a positive control.

NOTE 1: Mixed spectral results may be used for salt form determination or as a presumptive test for one or more compounds in the sample.

NOTE 2: Spectral subtraction may be used to fulfill the requirements for a confirmatory result. The spectrum must be labeled as a subtraction result. The original spectrum, the spectrum of the compound(s) being subtracted, and the final subtraction result must be included.

- For attenuated total reflectance (ATR), includes negative control data for a background spectrum collected with the ATR anvil up and a blank spectrum with the ATR anvil in contact with the stage.

NOTE: For composite analysis, one background is sufficient for each series of IR spectra collected provided a blank is obtained prior to each individual sample spectrum. For multi-unit analysis (pre-composite), refer to 2-5.1.

- Displays all spectra in the same units (i.e., transmittance, absorbance, or reflectance).
- Evaluates the data using the following acceptance criteria:
  - The overall sample spectral pattern (relative peak intensities and wavenumbers) corresponds to that of the positive control spectrum.
  - The observed wavenumbers for prominent, well-defined signals between 2000  $\text{cm}^{-1}$  and 650  $\text{cm}^{-1}$  in the sample spectrum are within 4  $\text{cm}^{-1}$  of those in the positive control spectrum.

NOTE: This correspondence may be demonstrated by displaying the measured wavenumbers on each spectra or by overlaying the sample and positive control spectra.

- The sample spectral pattern between 4000  $\text{cm}^{-1}$  and 2000  $\text{cm}^{-1}$  corresponds to that of the positive control spectrum.
  - No unexplainable extraneous signals are observed in the sample spectrum.
- Accepts results as confirmatory when the above criteria are met. Otherwise, results may be considered presumptive, provided the wavenumber acceptance criterion has been fulfilled.

#### 5.10.8 Raman Spectroscopy

The FC:

- Uses Raman as a confirmatory technique in the qualitative analysis of controlled and non-controlled substances.
- Uses Raman as a confirmatory test for identification purposes by comparing the spectrum of the sample to that of a positive control.

NOTE 1: Mixed spectral results may be used as a presumptive test for one or more compounds in the sample or for salt form determination.

NOTE 2: Spectral subtraction may be used to fulfill the requirements for a confirmatory result. The spectrum must be labeled as a subtraction result. The original spectrum, the spectrum of the compound(s) being subtracted, and the final subtraction result must be included.

- Evaluates the data using the following acceptance criteria:
  - The overall sample spectral pattern (relative peak intensities and Raman shifts) corresponds to that of the positive control spectrum.
  - The observed Raman shifts for prominent and well-defined signals in the sample spectrum are within 4 cm<sup>-1</sup> of those in the positive control spectrum.

NOTE: This correspondence may be demonstrated by displaying the shifts on each spectrum or by overlaying the sample and reference spectrum.

- No unexplainable extraneous signals are observed in the sample spectrum.
- Accepts results as confirmatory when the above criteria are met. Otherwise, results may be considered presumptive provided the Raman shift acceptance criterion has been fulfilled.

#### 5.10.9 Mass Spectrometry

The FC:

- Uses MS as either a confirmatory or a separation technique in the qualitative analysis of controlled and non-controlled substances.

##### 5.10.9.1 MS as Confirmatory

The FC:

- Uses MS as a confirmatory test for identification purposes by comparing the fragmentation spectrum of the sample to that of a positive control and by evaluating the data using the following acceptance criteria:
  - The overall sample fragmentation pattern (relative ion abundances, isotopic distributions, and *m/z* values) corresponds to that of the positive control spectrum.

NOTE: Relative ion abundance is measured with respect to the most intense signal in the spectrum.

- The measured *m/z* values for prominent ions in the sample spectrum are of the same nominal mass as those in the positive control spectrum.



- For EI-MS, if the majority of the sample spectrum is of low abundance, then the spectrum is expanded and re-evaluated against a similarly expanded positive control spectrum. Both the full and expanded spectra of both the sample and positive control must be shown.
- For high-resolution MS, the measured  $m/z$  values for prominent ions in the sample spectrum are within 5 ppm of the positive control spectrum values.
- The molecular ion must be observed in the sample spectrum if it is expected and observed in the positive control spectrum.
- No unexplainable extraneous ions are observed in the sample spectrum.
- Accepts results as confirmatory when the above criteria are met. Otherwise, results may be considered presumptive, provided the  $m/z$  acceptance criterion has been fulfilled.

#### 5.10.9.2 MS as Separation

The FC:

- Uses soft-ionization MS as a separation test when no fragmentation information is generated (i.e., only pseudo-molecular ions are observed). The pseudo-molecular ion, to include salt or solvent adduct signals, of the sample is compared to that of a positive control and evaluated using the following acceptance criteria:
  - For low-resolution MS, accepts the results when the measured  $m/z$  value in the sample spectrum is the same nominal mass as that of the positive control spectrum.
  - For high-resolution MS, accepts the results when the measured  $m/z$  value in the sample spectrum is within 5 ppm of the positive control spectrum value.
- Accepts results as presumptive when the above criteria are met.

#### 5.10.10 Nuclear Magnetic Resonance Spectroscopy

The FC:

- Uses NMR as a confirmatory technique in the qualitative analysis of controlled and non-controlled substances.
- Uses NMR as a confirmatory test for identification purposes by comparing the spectrum of the sample to that of a positive control acquired using the same solvent and internal standard (if used).
- Evaluates the data using the following acceptance criteria:
  - The overall sample spectral pattern (multiplicity, relative signal intensity, and chemical shifts) corresponds to that of the positive control spectrum.

- The measured chemical shifts for all signals in the sample spectrum are within 0.2 ppm ( $^1\text{H}$ -NMR) (with the exception of labile proton signals) and 2 ppm ( $^{13}\text{C}$ -NMR) of those in the positive control spectrum.

NOTE: For other NMR experiments, acceptance criteria must be established within the laboratory and approved by the LD.

- No unexplainable extraneous signals are observed in the sample spectrum.
- Accepts results as confirmatory when the above criteria are met. Otherwise results may be considered presumptive, provided the chemical shift acceptance criterion has been fulfilled.

## 6.0 Quantitative Analysis

### 6.1 General Requirements

REDACTED

The FC:

- REDACTED
- Quantitates exhibits which require the result for statutory considerations (i.e., methamphetamine, amphetamine, oxycodone, PCP, and hydrocodone).
- Does not routinely quantitate samples when the primary controlled substance is estimated to be present below the 1% level.
- Performs quantitative analyses using validated methods, as posted on the Office of Forensic Sciences Document Control Center (SFDCC).
  - When a particular instrumental technique (i.e., LC, GC, CE, NMR) is selected as the technique of choice, a standardized method must be used, if available.
  - When a standardized method is not available for the selected technique, a laboratory-validated method must be used.
  - Laboratory-validated methods are transferrable between instruments and laboratories, as long as the Level 2 validation requirements (as specified in 1-4.3.2) are fulfilled.
- Performs quantitative analysis of secondary controlled substance(s) when the secondary controlled substance(s) is estimated to be present at a level of 5% or greater, provided that a sufficient amount of sample is available.
  - If additional controlled substances appear to be present as the result of naturally occurring alkaloids, incomplete reactions or sample breakdown, quantitation of these substances is only necessary when they are the predominant controlled substance in the exhibit.
- Does not quantitate naturally occurring, active constituents in botanicals such as opium (e.g., morphine), peyote (e.g., mescaline), and mushrooms (e.g., psilocybin).

NOTE: Enforcement analyses performed by SFL1 are exempt from this requirement.

- Performs quantitative analysis of non-controlled substances, including listed chemicals at the discretion of the LD, or when necessary to support investigations.
- Bases all reported purities on reviewable data and observations.
  - Any data or observation which does not correlate with the quantitation results must be fully explained, or the quantitative value cannot be reported.

- Includes negative and positive controls (i.e., blanks and QC solutions) in the reviewable data.

## **6.2 Quantitative Procedures**

The FC:

- Uses only Class-A volumetric glassware or calibrated automatic pipettes for volume measurements associated with solution preparations.
- Applies minimum net weight requirements (2-3.1) to preparation of all quantitation solutions.
- Equilibrates solutions maintained under refrigeration to room temperature prior to use.
- Prepares negative controls (blanks) from the same solvent or internal standard solution used to prepare the quantitative sample.
  - A negative control sample is analyzed immediately prior to the first injection of each exhibit to ensure that the instrument and solvent are free from potential carry-over or contamination.

## **6.3 Calibrant Solutions**

### **6.3.1 Separation Methods**

The FC:

- Prepares a reference material (calibrant) solution using a certified reference material (CRM).
- Factors the documented purity of the CRM into the final concentrations of the calibrant solutions.
- Ensures that calibrant solutions are used for only one month from the date of preparation.

NOTE: Calibrant solutions may be maintained and used past one month provided they are checked for continued use. Checks shall demonstrate that the concentration of the target analyte remains within the acceptance limits. These checks must be conducted once a month and the results documented in a manual or electronic logbook. Supporting data must be maintained.

- Establishes a single-point calibration curve during the same sequence as the sample and by analyzing at least one injection of a calibrant solution, generally representing the middle of the validated working range according to 2-6.5.1, and using zero as the y-intercept.

NOTE: Laboratory-validated methods may be used in accordance with the original validation procedures (e.g., single or 3-point calibration curve).

### **6.3.2 qNMR Methods**

- Weighs and directly adds to the sample an internal standard (IS) with traceable purity.
  - Factors the documented purity of the IS into the final amount of the IS.

OR

- Prepares an internal standard solution (ISS) using an IS with traceable purity.
  - Factors the documented purity of the IS into the final concentration of the ISS.
  - Ensures that the ISS is used for only one month from the date of preparation.

NOTE: ISS may be maintained and used past one month provided it is checked for continued use. Checks shall demonstrate that the concentration of the target analyte remains within the acceptance limits. These checks must be conducted once a month and the results documented in a manual or electronic logbook. Supporting data must be maintained.

#### 6.4 Quality Control Samples

The FC:

- Uses a quality control (QC) sample from SFL1, when available, to prepare quality control (QC) solutions for use as positive controls during quantitative analysis.
  - Prepare QC samples for any substances not provided by SFL1 from the laboratory's reference materials collection or other traceable source (e.g., pharmaceuticals obtained from the manufacturer).
  - Prepare QC samples to include the analyte of interest and that mimic the approximate composition and purity of commonly encountered exhibits.

NOTE: With supervisory approval, a substitute analyte may be used for the NMR QC sample when only limited quantities of reference material are available.

- For separation techniques, prepares two solutions at target analyte concentrations that represent the lower and higher ends of the method's working range.
- For NMR quantitation, prepares one solution within the solubility limits of the method.
- Uses QC solutions until they are depleted or the results fall outside of the acceptance criteria. (See 2-6.6)
- Documents QC solution preparation procedures in the *Quantitation* test or attachments.

#### 6.5 Sample Preparation and Analysis

The FC:

- Prepares a solution(s) so that the target analyte concentration is bracketed between the high and low QC solutions concentrations.
- For powders, crystalline materials, and solid dosage forms, obtains the test sample amount from the composite as specified below.

- If a representative composite was prepared but minimum test amounts are not used, regardless of exhibit's net weight, obtain *Supervisory Approval* and use the Scenario I purity statement on the DEA-113 (refer to Appendix 2B).
- For moist composites amenable to grinding but not sieving, a representative test amount is at least 100 mg. Use the Scenario H purity statement on the DEA-113 (refer to Appendix 2B).

Powders, Crystalline Materials, and Solid Dosage Fo
<i>Total Net Weight &lt; 100 mg:</i> <ul style="list-style-type: none"> <li>• Purity testing is not normally performed unless requested by the customer</li> </ul>
<i>Total Net Weight ≥ 100 mg:</i> <ul style="list-style-type: none"> <li>• Refer to Table 2</li> </ul>

NOTE: Minimum amounts listed in Table 2 ensure the analytical sample used for quantitation is representative of the composite.

- For liquids and solutions, obtains the test sample from the composite as specified below.
  - Use the Scenario H or Scenario J purity statements on the DEA-113 (refer to Appendix 2B)

Liquids/Solutions
<i>Total Volume &lt; 5 mL:</i> <ul style="list-style-type: none"> <li>• Purity testing is not normally performed unless requested by the customer</li> </ul>
<i>Total Volume ≥ 5 mL:</i> <ul style="list-style-type: none"> <li>• Minimum 1.0 mL</li> </ul>

- For gummy samples, other forms, and exhibits for which no composite is prepared, obtains the test sample by combining multiple independent portions from the exhibit.
  - Use the Scenario J purity statement on the DEA-113 (refer to Appendix 2B).

Gummy
<p><i>&lt;5.0 g per Unit:</i></p> <ul style="list-style-type: none"> <li>Purity testing is not normally performed unless requested by the customer</li> </ul>
<p><i>≥ 5.0 g per Unit:</i></p> <ul style="list-style-type: none"> <li>≥ 0.5 g (sample portions from various randomly selected units)</li> </ul>

Other Forms/No Composite Prepared
Purity testing is not normally performed unless requested by the customer

#### 6.5.1 Performing a Quantitative Analysis Using a Separation Method

The FC:

- Quantitates an unknown sample using a minimum of the calibrant, a negative control, the sample injected in duplicate, and a set of bracketing high and low concentration QC solutions.
  - The QC solutions must bracket the unknown sample in concentration (high and low) and in time run in the sequence (before and after).

NOTE 1: Multiple exhibits may be analyzed between bracketing QC solutions.

NOTE 2: Blanks between multiple injections of the same preparation are not necessary.

#### 6.5.2 Performing a Quantitative Analysis Using qNMR

The FC:

- Quantitates an unknown sample using a minimum of a blank, a sample, and a single QC solution.
  - A daily NMR blank and a daily QC solution must be associated with each batch of samples. Multiple batches can use the same daily blank and QC solutions. The NMR blank and sample must be prepared from the same deuterated solvent source.

#### 6.6 Acceptance Criteria

The FC ensures:

- Blanks are free of carry-over and contamination.
- QC solutions are within  $\pm 5\%$  relative to the known prepared purity of the QC sample.

- For separation techniques, the RSD of the response ( $A_{Spl}$  or  $A_{Spl}/A_{IntStd}$ ) for the duplicate injections of the unknown sample is less than 2%.
- qNMR results are derived from an average of at least three acceptable integrals.
  - The use of fewer than three integrals must be supported by the peak height quantitation information and approved by the supervisor.
- The maximum acceptable quantitative result is 104%.

## 6.7 Reporting Quantitation Results

The FC:

- Reports the final purity value as the average of the injections analyzed, provided that the RSD criteria is fulfilled (2-6.6).
- Reports the final purity value for NMR as the average of multiple runs (multiple integrals) of the sample preparation.
- Obtains a final purity value as follows if two or more sample preparations or instrumental techniques are used:
  - Average the purity results obtained for each individual preparation/technique and document each individual average in the Case Details Report (CDR) or attachments.
  - Combine averaged purity results only if each individual averaged result falls within the UME associated with the mean of all averages.
  - Document any results that are outside of the acceptance criteria.
  - The final reported purity result is the mean of all accepted averages.
- Reports controlled substances in decreasing order of abundance (if known).
- Calculates and reports the quantitative result as the predominant salt form (if known).
  - When the salt form is unknown, document the salt form used to calculate the reported quantitative value, e.g., heroin (calculated as hydrochloride).
- Reports the final purity result in percentage truncated to match the significance of the final reported uncertainty.
- Reports purity results greater than 100% (e.g., 100.6%) as 100%.
- Calculates and reports the purity UME per 2-7.



## 7.0 Determining the Uncertainty of Measurement Estimates for Quantitative Values

The FC:

- Calculates the uncertainty of measurement estimate (UME) associated with quantitative values per Appendix 2F.
- Reports the UME for the purity of all quantitated substances on the DEA-113.

### 7.1 Reporting UME

The FC:

- Calculates all UMEs using the final averaged % purity value (prior to truncation).
- Rounds the final expanded UME to one significant figure using ISO/NIST rounding rules:
  - When the digit following the one to be retained is less than five, keep the retained figure unchanged. Example: To one significant figure, 2.441 becomes 2.
  - When the digit following the one to be retained is greater than five, increase the retained figure by one. Example: To one significant figure, 2.773 becomes 3.
  - When the digit following the one to be retained is five and at least one of the following digits is greater than 0, increase the retained figure by one. Example: To one significant figure, 0.4507 becomes 0.5.
  - When the digit following the one to be retained is five and all of the following digits are 0, keep the retained figure unchanged if it is even or increase by one if it is odd. Examples: To one significant figure, 3.500 becomes 4, and 4.500 becomes 4 (the final digit is always even).
- Documents UMEs in LIMS and on the DEA-113.
- Includes a statement in the *Observations, Results, and Conclusions* section of the DEA-113 when purity results are reported (refer to Appendix 2B, Scenarios H, I, or J).
- Includes a copy of the completed *Uncertainty Calculator* in LIMS (SFL1 only).

### 7.2 Revising UMEs

The Office of Forensic Sciences Quality Assurance Section (SFQ):

- Reviews and updates the uncertainty estimates for purity determinations every accreditation cycle using cumulative system-wide Proficiency Testing Program (PTP) results and other collaborative data.

## 8.0 Processing Evidence After Analysis

The FC:

- Determines reserve weight of exhibit.
- Reseals reserve evidence as described below.
- Returns completed evidence in accordance with the Laboratory Operations Manual (LOM) 73.

### 8.1 Resealing Evidence: Plastic Sealed Evidence Envelopes

The FC:

- Reseals the evidence in the same interior packaging or substitute container(s) (as appropriate) to prevent spilling of the inner contents and to allow for visual examination of the interior packaging and contents.
  - For evidence not composited following Option 1, repackage evidence in a minimum of three populations – composite, remaining portion from analyzed units, and unanalyzed units.
  - In the case of a residue sample with insufficient material for two samplings, include the procedural blank(s) and sample vial(s) used in the analysis of the exhibit.
- Places the packaging or containers into the original evidence envelope.
- Prepares an official DEA evidence seal bearing the sealing FC's signature, the date of sealing, *IA Case Number*, *IA Exhibit Number*, and *LIMS Case Number*.
- Affixes this seal on the outside of the evidence envelope at the end requiring the seal, at the approximate center, parallel with the opening in the bottom of the envelope, and on the same side of the envelope as the label.
  - Seal individual sub-exhibits in the same evidence envelope.
- Heat-seals the open end of the evidence envelope through the affixed seal.
- Inspects the integrity of the heat seal.
- Obtains the gross weight after analysis of each individual sealed evidence envelope.
- Reports the total gross weight after analysis in LIMS.
- Records a description of the reserve evidence and the date resealed in LIMS.

NOTE: Descriptions that are too long to fit in the LIMS field may be added as an attachment to the test (as PDF or image files).

- At a minimum, records the gross weight after analysis and the date resealed on the evidence envelope label.

## **8.2 Resealing Evidence: Other Evidence Containers**

The FC:

- Reseals the evidence in the same interior packaging or substitute container(s) to prevent spilling of the inner contents and to allow for visual examination of the interior packaging and contents.
  - For evidence not composited following Option 1, repackage evidence in a minimum of three populations – composite, remaining portion from analyzed units, and unanalyzed units.
- Reseals evidence retained in opaque containers in the following manner:
  - Seal all interior evidence within transparent containers (PSEEs) with an official DEA evidence seal.

NOTE: Only applicable to threshold portion for DEA/DOJ submissions. For other agencies, this only applies to units opened for analysis.

- Weigh each sealed interior package or container using the balance software.
  - Record the weight and the date sealed on the package or container.
  - Document the weights in LIMS.
- Places the interior package(s) or container(s) into the original evidence container(s) (e.g., boxes, suitcases, etc.).

NOTE: Add filler material, as necessary, to prevent the contents from shifting.

- Reseals the exterior evidence container(s) with fiber-reinforced tape by completely encircling the container in two directions.
- Places an evidence seal bearing the FC's signature, the date of sealing, IA Case Number, IA Exhibit Number, and LIMS Case Number at the junction where the tape ends meet while also adhering part of the evidence seal to the actual container.
- Obtains the gross weight of each evidence container.
- Reports the total gross weight after analysis in LIMS.
- Records a description of the reserve evidence and the date resealed in LIMS.

NOTE: Descriptions that are too long to fit in the LIMS field may be added as an attachment to the test (as PDF or image files).

- At a minimum, records the gross weight after analysis and the date resealed on the evidence label.

### **8.3 Resealing Bulk Exhibits**

#### **8.3.1 Submissions by DEA and Other Department of Justice Agencies**

The FC:

- Separates the exhibit into two portions: a threshold amount for retention and a bulk portion for destruction.
  - The threshold portion consists of the reserve composite, along with a sufficient amount of non-composite material to meet the required threshold weight.

NOTE: For exhibits comprised of multiple multi-layer units, it is not necessary to split unopened units to meet the specified threshold.
  - The bulk portion consists of the remaining material.
- Places the threshold portion and at least one empty original packaging (if possible) into a single, original or substitute evidence container.
  - If a substitute container is used, maintain the original evidence label in the new container or photograph the original evidence label and attach the photograph to the case file.
- Places the bulk portion and all unretained empty packaging into remaining original or substitute evidence containers.
- Reseals the exterior evidence containers as in 2-8.1 or 2-8.2, as appropriate.
  - Wrap the threshold evidence container with red tape.
  - Mark the evidence container(s) as appropriate (i.e., “threshold” or “bulk”).
- Selects *Organize My Work, Evidence Containers*.
- Changes the *Container Code* for the threshold amount to “Threshold” and the bulk portion to “Bulk Evidence.”
- Prints the evidence container labels with the new container code designations.
- Affixes the new labels over the existing container labels.
- Obtains the gross weight of each evidence container.
- Reports the total gross weight after analysis in LIMS.
- Records a description of the reserve evidence and the date resealed in LIMS.

NOTE: Descriptions that are too long to fit in the LIMS field may be added as an attachment to the test (as PDF or image files).

- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- Includes the following statement in the *Remarks* of the *Evidence Details* section of the DEA-113:
  - For DEA evidence, annotate the amount of bulk evidence, along with the intent of destruction.  
“\_\_\_ gram(s) held for destruction pending written notification.”
  - For bulk evidence received from other Department of Justice agencies, annotate the amount separated in excess of threshold.  
“\_\_\_ gram(s) separated in excess of the threshold.”

### 8.3.2 Submissions by Other Agencies

The FC:

- Places the reserve evidence into the original evidence container(s).
- NOTE: The same number of containers submitted by DHS should be returned (i.e., a representative sample is not to be separated from the bulk material).
- Reseals the exterior evidence container(s) as in 2-8.1 or 2-8.2, as appropriate.
  - Obtains the gross weight of each individual evidence container.
  - Reports the total gross weight after analysis in LIMS.
  - Records a description of the reserve evidence and the date resealed in LIMS.

NOTE: Descriptions that are too long to fit in the LIMS field may be added as an attachment to the test (as PDF or image files).

- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).

### 8.4 Creating Additional/New Evidence Containers

The FC:

- Repackages evidence using new or additional evidence containers as needed.
- Creates additional/new evidence container(s) in LIMS.
- Completes the lab exhibit description as appropriate.

- Prints container labels for the newly created evidence container(s).
- Affixes the label(s) to the new evidence container(s).
- Adds the *Additional Evidence Unit* test to each newly created exhibit.

## 9.0 Reanalyzing Evidence

- Contact the Office of Forensic Sciences Laboratory Management and Operations Section (SFM) for specific procedures for any scenario not covered below.

### 9.1 Reanalyzing DEA Exhibits Originally Analyzed in LIMS

The SC:

- Reopens the exhibit in LIMS.

NOTE: Return exhibit from storage, if applicable.

- Reroutes the exhibit to Chemistry.
- Sends the exhibit for analysis.
- Assigns the exhibit to the FC.

#### 9.1.1 Reanalysis

The FC:

- Makes no changes to the original analysis documentation in LIMS.
- Creates a new sub-exhibit in LIMS
- Names *Lab Exhibit* as “*Exhibit Number-R*” (e.g., 1-R, 1.01-R, 1A-K-R, 1B1-R, etc.).
- Documents as “*Reanalysis of Exhibit X*”, where X is the original exhibit number (e.g., 1, 1.01, 1A-K, 1B1).
- Adds all tests required for reanalysis to the newly created sub-exhibit.

NOTE: This includes the *Gross Weight*, *Other Notes*, and *Summary of Findings* tests.

- Obtains the gross weight of the sealed evidence using the balance software.
  - Use a substitute code, not the original exhibit’s barcode, when obtaining the gross weight.
- Records the gross weight and manually attaches the balance data to the *Gross Weight* test in the newly created sub-exhibit.
- Reopens the exhibit and records the date reopened in the appropriate section on the evidence container label and in LIMS.
- Performs the reanalysis.

- Annotates the reason for reanalysis in the *Other Notes* test.
- Reseals the evidence as described in 2-8.1 or 2-8.2.
- Records a description of the reserve evidence and the date resealed in LIMS.
- Obtains the gross weight after analysis.
  - Use a substitute code, not the original exhibit's barcode, when obtaining the gross weight after analysis.
- Records the gross weight after analysis and manually attaches the balance data to the *Gross Weight After Analysis* test in the newly created sub-exhibit.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).

### 9.1.2 Reporting

The FC:

- Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- Includes the following statement in the *Remarks of the Observations, Results, and Conclusions* section of the DEA-113:  
  
“*Supplemental report to reflect reanalysis. Refer to original laboratory report dated mm/dd/yyyy.*”

## 9.2 Reanalyzing DEA Exhibits Not Originally Analyzed in LIMS

The SC:

- Adds the DEA-7 and original DEA-113 to *Case Attachments*.
- Reopens the exhibit in LIMS.  
  
NOTE: Return exhibit from storage, if applicable.
- Renames *Lab Exhibit* as “*Exhibit Number-R*” (e.g., 1-R, 1.01-R, 1A-K-R, 1B1-R, etc.).
- Reroutes the exhibit to Chemistry.
- Sends the exhibit for analysis.
- Assigns the exhibit to the FC.



### 9.2.1 Reanalysis

The FC:

- Adds tests required for reanalysis of the exhibit, including the *Other Notes* test.
- Obtains the gross weight of the sealed evidence using the balance software.
- Reopens the exhibit and records the date reopened in the appropriate section on the evidence container label and in LIMS.
- Performs the reanalysis.
- Annotates the reason for reanalysis in the *Other Notes* test.
- Reseals the evidence as described in 2-8.1 or 2-8.2.
- Records a description of the reserve evidence and the date resealed in LIMS.
- Obtains the gross weight after analysis using the balance software.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).

### 9.2.2 Reporting

The FC:

- Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- Includes the following statement in the *Remarks of the Observations, Results, and Conclusions* section of the DEA-113:  
  
*“Supplemental report to reflect reanalysis. Refer to original laboratory report dated mm/dd/yyyy.”*

### 9.3 Reanalyzing Non-DEA Exhibits

- Refer to 2-9.1 for reanalysis of exhibits that have not yet been returned to the submitting agency.

The ES:

- Accepts resubmitted evidence into the laboratory.
- Creates a new *IA Exhibit* in LIMS.
- Enters the *IA Exhibit* as “*Exhibit Number-R*” (e.g., 1-R, 1.01-R, 1A-K-R, 1B1-R, etc.).

The SC:

- Adds the DEA-7 and original DEA-113 to *Case Attachments*, if original analysis was not done using LIMS.
- Assigns the exhibit to the FC.

### 9.3.1 Reanalysis

The FC:

- Adds tests required for reanalysis of the exhibit, including the *Other Notes* test.
- Obtains the gross weight of the sealed evidence.
- Reopens the exhibit and records the date reopened in the appropriate section on the evidence container label and in LIMS.
- Performs the reanalysis.
- Annotates the reason for reanalysis in the *Other Notes* test.
- Reseals the evidence as described in 2-8.1 or 2-8.2.
- Obtains the gross weight after analysis.
- Records a description of the reserve evidence and the date resealed in LIMS.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).

### 9.3.2 Reporting

The FC:

- Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- Includes one of the following statements in the *Remarks of the Observations, Results, and Conclusions* section of the DEA-113:

*"Supplemental report to reflect reanalysis. Refer to original laboratory report (original LIMS Case Number XXXX-SFLX-XXXXX) dated mm/dd/yyyy."*

OR

*"Supplemental report to reflect reanalysis. Refer to original laboratory report (original lab number XXXXXX) dated mm/dd/yyyy."*

## 10.0 Removing Sample for Defense Analysis, Reweighing Evidence, and Assessing Evidence Returned from Court

The FC:

- Follows directives as specified in the request documentation (e.g., court order, written agreement, etc.).
- Removes sample(s) for defense analysis as described below.
- Reweighs evidence as described below.
- Assesses evidence returned from court as described below.

### 10.1 Removing Sample(s) for Defense Analysis from DEA Exhibits Originally Analyzed in LIMS

The SC:

- Reopens the exhibit in LIMS.  
NOTE: Return exhibit from storage, if applicable.
- Adds applicable documentation (e.g., court order, etc.) to *Case Attachments*.
- Reroutes the exhibit to Chemistry.
- Sends the exhibit for analysis.
- Assigns the exhibit to the FC.

#### 10.1.1 Removing Sample(s)

The FC:

- Makes no changes to the original analysis documentation in LIMS.  
NOTE: *Reserve Weight* and *Gross Weight After Analysis* tests will be updated, but original balance data is not to be deleted.
- Documents all observations and measurements using the *Other Notes* test in LIMS, a DEA-86 or DEA-86a.
- Reopens the following LIMS tests:
  - *Reserve Weight*
  - *Description of Reserve Evidence*

- *Gross Weight After Analysis*
- *Summary of Findings*
- Adds the following LIMS tests:
  - *Exemplar Weight Removed*
  - *Other Notes*
- Obtains the gross weight of the sealed evidence using the balance software.
  - Use a substitute code, not the original exhibit's barcode, when obtaining the gross weight.
- Manually attaches the gross weight balance data to the *Other Notes* test.
- Reopens the exhibit and records the date reopened on the evidence container label and in the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Removes, or observes the removal of, the sample(s) for defense analysis and obtains a new reserve weight of the original exhibit.
  - Use the original exhibit's weight barcode so that the weight value and balance data automatically populate the *Reserve Weight* test in LIMS.
- Annotates the reason for sample removal (e.g., sample removed for defense analysis) in the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Creates a DFA unit in LIMS.
  - Use the exhibit number (e.g., 1, 1.01, 1A-K, 1B1, etc.) in the *Lab Exhibit* field.
  - Annotate the *Description* as "Defense Analysis of Exhibit X", where X is the exhibit number (e.g., 1, 1.01, 1A-K, 1B1).
  - Select *Place in New Container*.
  - Select the *Container Code* of "Defense Analysis" for the newly created container(s).
  - Print a container label(s) for the newly created container(s).
  - Affix the label to the new container(s).
  - Add and complete the *Other SP Sample Weight*, *Description of Reserve Evidence*, and *Gross Weight - SP/LP* test to the newly created unit.
- Reseals the evidence as described in 2-8.1 or 2-8.2.

- Records a description of the reserve evidence and the date resealed in the original *Description of Reserve Evidence* test in LIMS.

NOTE: The additional information is added without changing the original description.

- Obtains the gross weight after analysis using the balance software.
  - Use the original exhibit's barcode so that the weight value and balance data automatically populate the *Gross Weight After Analysis* test in LIMS.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- Adds a copy of the DEA-86 or DEA-86a to the *Other Notes* test.
- Returns evidence (original and defense sample) to vault.

#### 10.1.2 Reporting

The FC:

- Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
- Ensures the supplemental report reflects the new reserve weight.
- Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:  
"\_\_\_\_\_ grams removed for defense analysis."
- Includes the following statement in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113:  
"Supplemental report to reflect removal of sample for defense analysis and revised reserve weight. Refer to original laboratory report dated mm/dd/yyyy."

#### 10.2 Removing Samples(s) for Defense Analysis from DEA Exhibits Not Originally Analyzed in LIMS

The SC:

- Adds the DEA-7 and original DEA-113 to *Case Attachments*.
- Reopens the exhibit in LIMS.

NOTE: Return exhibit from storage, if applicable.

- Adds applicable documentation (e.g., court order, etc.) to *Case Attachments*.

- Reroutes the exhibit to Chemistry.
- Sends the exhibit for analysis.
- Assigns the exhibit to the FC.

#### 10.2.1 Removing sample(s)

The FC:

- Documents all observations and measurements using the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Adds the following LIMS tests:
  - *Gross Weight*
  - *Exemplar Weight Removed*
  - *Other Notes*
  - *Reserve Weight*
  - *Description of Reserve Evidence*
  - *Gross Weight After Analysis*
- Obtains the gross weight of the sealed evidence using the balance software.
- Records the gross weight on the evidence label.
- Reopens the exhibit and records the date reopened on the evidence container label.
- Removes, or observes the removal of, the sample(s) for defense analysis and obtains a new reserve weight of the original exhibit.
- Annotates the reason for sample removal in the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Creates a DFA unit in LIMS.
  - Use the exhibit number (e.g., 1, 1.01, 1A-K, 1B1, etc.) in the *Lab Exhibit* field.
  - Annotate the *Description* as “*Defense Analysis of Exhibit X*”, where X is the exhibit number (e.g., 1, 1.01, 1A-K, 1B1).
  - Select *Place in New Container*.
  - Select the *Container Code* of “*Defense Analysis*” for the newly created container(s).

- Print a container label(s) for the newly created container(s).
- Affix the label to the new container(s).
- Add and complete the *Other SP Sample Weight, Description of Reserve Evidence, and Gross Weight - SP/LP* test to the newly created unit.
- Reseals the evidence as described in 2-8.1 or 2-8.2.
- Records a description of the reserve evidence and the date resealed in LIMS.
- Obtains the gross weight after analysis using the balance software.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- Adds a copy of the DEA-86 or DEA-86a to the *Other Notes* test.
- Returns evidence (original and defense sample) to vault.

#### 10.2.2 Reporting

The FC:

- Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
  - Use the appropriate non-LIMS DEA-113 on SFDCC.
- Adds original analysis results to the non-LIMS DEA-113.
- Ensures the supplemental report reflects the new reserve weight.
- Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:  
  
"\_\_\_\_\_ grams removed for defense analysis."
- Includes the following statement in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113:  
  
"Supplemental report to reflect removal of sample for defense analysis and revised reserve weight. Refer to original laboratory report dated mm/dd/yyyy."
- Prints, signs, and submits the supplemental report to the SC.

The SC:

- Adds approved non-LIMS supplemental report to *Case Attachments*.

- Sends non-LIMS supplemental report to case agent.
- Attaches copy of email communication with case agent to *Case Attachments*.

### **10.3 Removing Samples(s) for Defense Analysis from Non-DEA Exhibits**

- Refer to 2-10.1 for removal of sample for defense analysis for exhibits that have not yet been returned to the submitting agency.

The ES:

- Accepts resubmitted evidence into the laboratory.
- Creates a new *IA Exhibit* in LIMS.
- Enters the *IA Exhibit* as "*Exhibit Number-D*" (e.g., 1-D, 1.01-D, 1A-K-D, 1B1-D, etc.).

The SC:

- Adds the DEA-7 and original DEA-113 to *Case Attachments*, if original analysis was not done using LIMS.
- Adds applicable documentation (e.g., court order, etc.) to *Case Attachments*.
- Assigns the exhibit to the FC.

#### **10.3.1 Removing sample(s)**

The FC:

- Documents all observations and measurements using the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Adds the following LIMS tests:
  - *Gross Weight*
  - *Exemplar Weight Removed*
  - *Other Notes*
  - *Reserve Weight*
  - *Description of Reserve Evidence*
  - *Gross Weight After Analysis*
- Obtains the gross weight of the sealed evidence using the balance software.



- Reopens the exhibit and records the date reopened on the evidence container label.
- Removes, or observes the removal of, the sample(s) for defense analysis and obtains a new reserve weight of the original exhibit.
- Annotates the reason for sample removal in the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Creates a DFA unit in LIMS.
  - Use the new exhibit number (e.g., 1-D, 1.01-D, 1A-K-D, 1B1-D, etc.) in the *Lab Exhibit* field.
  - Annotate the *Description* as “*Defense Analysis of Exhibit X*”, where X is the original exhibit number (e.g., 1, 1.01, 1A-K, 1B1).
  - Select *Place in New Container*.
  - Select the *Container Code* of “*Defense Analysis*” for the newly created container(s).
  - Print a container label(s) for the newly created container(s).
  - Affix the label to the new container(s).
  - Add and complete the *Other SP Sample Weight, Description of Reserve Evidence, and Gross Weight - SP/LP* test to the newly created unit.
- Reseals the evidence as described in 2-8.1 or 2-8.2.
- Records a description of the reserve evidence and the date resealed in LIMS.
- Obtains the gross weight after analysis using the balance software.
- At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- Adds a copy of the DEA-86 or DEA-86a to the *Other Notes* test.
- Returns evidence (original and defense sample) to vault.

### 10.3.2 Reporting

The FC:

- Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
  - Use the appropriate non-LIMS DEA-113 on SFDCC.
- Adds original analysis results to non-LIMS DEA-113.

- Ensures the supplemental report reflects the new reserve weight.
- Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:  
"\_\_\_\_\_ grams removed for defense analysis."
- Includes the following statement in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113:

*"Supplemental report to reflect removal of sample for defense analysis and revised reserve weight. Refer to original laboratory report (original LIMS Case Number XXXX-SFLX-XXXXX) dated mm/dd/yyyy."*

OR

*"Supplemental report to reflect removal of sample for defense analysis and revised reserve weight. Refer to original laboratory report (original lab number XXXXXX) dated mm/dd/yyyy."*

- Prints, signs, and submits the supplemental report to the SC.

The SC:

- Adds approved non-LIMS supplemental report to *Case Attachments*.
- Sends non-LIMS supplemental report to case agent.
- Attaches copy of email communication with case agent to *Case Attachments*.

#### 10.4 Reweighing Evidence

The SC:

- Reopens the exhibit in LIMS.  
  
NOTE: Return exhibit from storage, if applicable.
- Adds applicable documentation (e.g., court order, etc.) to *Case Attachments*.
- Reroutes the exhibit to Chemistry.
- Sends the exhibit for analysis.
- Assigns the exhibit to the FC.

The FC:

- Makes no changes to the original analysis documentation in LIMS.
- Adds the *Other Notes* test to the exhibit.

- Documents all observations and measurements using the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- Obtains all applicable weights.
- Adds a copy of the DEA-86 or DEA-86a to the *Other Notes* test.
- Manually attaches the weight data (if using the balance software) to the *Other Notes* test.
- Reseals the evidence as described in 2-8.1 or 2-8.2.

#### **10.5 Assessing Evidence Returned from Court**

- Evidence returned from court shall be assessed in accordance with LOM 73.

## 11.0 The Analytical Record

- SFL1 maintains the analytical record as a paper case file (i.e., all raw data, observations, and calculations).

### 11.1 General Instructions

The FC:

- Uses the LIMS tests to record all raw data, observations, and calculations at the time they are made.
- Documents results so that they are identifiable to a specific task and in a manner that permits adequate reconstruction of the analysis or examination performed.
- Documents the qualitative method used.
- Documents the quantitative method used.
- Documents the DEA property inventory numbers of all equipment and instruments used in the *Equipment* tab of the appropriate test.
- Documents the basic parameters/conditions for all instruments used in the appropriate test or on the attachments (e.g., spectra, chromatograms, etc.).
- Captures all weighing events using the balance software.
- Reports all weights, quantitation results, and uncertainties to the appropriate number of significant figures.
- Attaches photos or digital images to the specific test in LIMS.

NOTE: If the photo or digital image does not relate to a specific LIMS test, then attach to the *Image* finding of the *Description of Exhibit and Sampling* test.

- Records reference material unique identifier(s) in the appropriate test or on the attachments.
- Obtains a witness to verify an annotation or correction related to a discrepancy on any evidence-related document (e.g., weight discrepancies, evidence description discrepancies).
  - The person verifying the discrepancy electronically witnesses with one's username and password, in the appropriate test where the correction or annotation is needed.

### 11.2 Completing LIMS Tests

The FC:

- For routine analysis, adds, at a minimum, the following LIMS tests:

- *Gross Weight*
- *Description of Evidence*
- *Description of Exhibit and Sampling*
- *Net Weight*
- *Reserve Weight*
- *Gross Weight After Analysis*
- *Description of Reserve Evidence*
- *Summary of Findings*
- For sub-exhibit analysis, adds, at a minimum, the following LIMS tests:
  - *Description of Exhibit and Sampling*
  - *Net Weight*
  - *Reserve Weight*
- For REDACTED Latent Print units, adds the following LIMS tests:
  - *Applicable SP Weight Test*
  - *Description of Reserve Evidence*
  - *Gross Weight After Analysis (SP/LP)*
- For No Analysis exhibits in the possession of the FC, adds, at a minimum, the following LIMS tests:
  - When seals are intact:
    - *Gross Weight*
    - *No Analysis Performed*
    - *Summary of Findings*
  - When seals are broken:
    - *Gross Weight*
    - *Description of Evidence*

- *No Analysis Performed*
- *Description of Reserve Evidence*
- *Gross Weight After Analysis*
- *Summary of Findings*
- The remark “*No Analysis as per [Insert Reason]*” is added to the *Observations, Results, and Conclusions* section of the DEA-113.
- For exhibits re-opened, updates the following tests, as applicable:
  - *Description of Evidence*
  - *Reserve Weight*
  - *Gross Weight After Analysis*
  - *Description of Reserve Evidence*
  - *Summary of Findings*
- Completes all LIMS tests as described in Appendix 2F.

The SC:

- For an exhibit which does not require analysis and is not in the possession of a FC, selects *Return to CR through Cases Ready for Assignment* alert.
- Sends to storage, after status change to *CR In-Processing*, in the *Exhibits* tab of *Case Management*.
- Selects *Storage Only* and adds reason in the *Comments* field.
- Sends report to the case agent through the *Case Attachments* tab in *Case Management* (these reports will not appear in *Reports Pending Delivery*).

### 11.3 Supporting Data

The FC:

- Includes spectral, chromatographic, and other instrumental data in the LIMS case file.
- Ensures that each item of data is annotated, at minimum, with:
  - A unique identifier
  - Date and time of analysis

#### 11.4 The Laboratory Report (DEA-113) – General Information

The FC:

- Prepares a DEA-113 to report results for all analyzed evidence and proficiency testing samples.
- Prepares a DEA-113 to document when there was “*No Analysis Performed.*”
- Prepares a DEA-113 to document when there was an “*Inconclusive*” result.
  - Use the following statement in the *Remarks* regardless of the number of units – “*Identification pending further analysis.*” Other identified substances in the exhibit are reported per Appendix 2B.
- Ensures that LIMS automatically populates all fields on the DEA-113, except the *Remarks* fields. (See 2-11.5)
- Reports identified substances in the following order:
  - Controlled substances in order of abundance, if known, regardless of whether a substance was quantitated.
  - Non-controlled substances in order of abundance, if known.
- Reports reserve weights (non-exemplar exhibits) in the same units as the net weight and according to the following rules:
  - If the *raw* RW has more decimal places than the *reported* NW, truncate RW to same number of decimal places as *reported* NW.

*Example:*

If *reported* NW = 123.4 g and *raw* RW=122.125 g; then truncate RW to 122.1 g
  - If the *raw* RW has same decimal places as the *reported* NW, leave RW as is.

*Example:*

If *reported* NW = 13.4 kg and *raw* RW=13.2 kg; then leave RW as 13.2 kg
  - If the *raw* RW has fewer decimal places than the *reported* NW, leave RW as is.

*Example:*

If *reported* NW = 83.425 g and *raw* RW=83.2 g; then leave RW as 83.2 g
- Reports gross weights, net weight (exemplars), reserve weight (exemplars), separated bulk weights, and REDACTED as follows:

- Weight < 10 g – truncate and report to two significant figures (e.g., 0.86 g, 6.7 g)
- $10 \leq \text{Weight} < 1000 \text{ g}$  – truncate and report to tenth of a gram (e.g., 96.2 g, 711.0 g)
- Weight  $\geq 1000 \text{ g}$  – truncate and report to four significant figures (e.g., 2013 g, 327.6 kg)
- Reports volumes (net and reserve) as follows:
  - Less than 1 mL: Volume not reported.
  - Less than 100 mL: Truncate and report to one decimal place (e.g., 9.3 mL, 85.3 mL).
  - $100 \leq \text{Volume} < 1000 \text{ mL}$ : Truncate and report to whole (e.g., 325 mL).
  - Volume  $\geq 1000 \text{ mL}$ : Truncate and report to four significant figures (e.g., 2013 mL, 327.6 L).
- Report dosage units (net and reserve; tablets, capsules, impregnated paper) as follows:
  - Less than 1 dosage unit: Dosage units not reported.
  - 1-99 dosage units: Report to whole, if counted (e.g., 50 tablets); report to one decimal place, truncated, if calculated or extrapolated (e.g., 7.2 capsules, 88.6 tablets).
  - 100 or more dosage units: Report to truncated whole value, if counted, calculated or extrapolated (e.g., 325 capsules).
- Calculates purity equivalencies as follows:
  - Tablets/Capsules: Multiply the final (truncated) % purity by the average weight per unit.
  - Liquids: Multiply the final (truncated) % purity by the density of the liquid.
- Truncates and reports purity equivalencies as whole numbers if result is  $\geq 10$ , or two significant figures if result is < 10. For example,

Drug Form	Analysis Result	Reported Result
<b>Tablets/Capsules:</b>	9.56 mg/tablet	9.5 mg/tablet
	42.56 mg/tablet	42 mg/tablet
	125.25 mg/capsule	125 mg/capsule
<b>Liquids:</b>	321.123 mg/mL	321 mg/mL
	85.642 mg/mL	85 mg/mL
	8.456 mg/mL	8.4 mg/mL
<b>Other:</b>	34.78 µg/dose	34 µg/dose

- Submits completed DEA-113 for review.



#### 11.4.1 The Laboratory Report (SFL1 only)

The FC:

- Prepares a DEA-113 to report results for all analyzed enforcement evidence and proficiency testing samples.
- REDACTED
- Reports results of analysis from foreign operations per 2-11.7.
- Prepares a DEA-113 to document when there was “*No Analysis Performed*” on enforcement evidence.
- Submits completed DEA-113 for review.

#### 11.5 The Laboratory Report (DEA-113) – Remarks

The FC:

- Utilizes standardized statements available through the “*Insert Phrase*” options in *Examiner Reports Management*.
- Obtains supervisory approval prior to inserting non-standardized statements.
- Enters statements in the *Observations, Results, and Conclusions* section to document the following, as applicable:
  - Procedure for net weight determination
  - Net weight uncertainty statement
  - Total unit count and volume
  - Purity, when determined, and uncertainty statements
  - Purity equivalencies, when determined (e.g., mg/unit or mg/mL)
  - Remarks for *No analysis, Storage only, Supplemental, or Amended reports*
  - Department of Justice (DOJ) Uniform Language for Testimony and Reports (ULTR) reference statement (refer to Appendix 2B)
- Enters statements in the *Exhibit Details* section to document the following, as applicable:
  - REDACTED
  - Bulk evidence separation

- Latent print evidence separation
- Packaging and gross form descriptions with an *Other* finding
- Explanations of abbreviations or terminology used on the DEA-113
- Enters statements in the *Exhibit Analysis* section to document the following, as applicable:
  - Sampling procedure for qualitative analysis (statistical or non-statistical)
  - Qualifying statements for the reported identification(s)
- Enters statements in the *Certifications* section, as applicable:
  - Certificate of compliance statements

#### 11.6 Reviewing Analysis and Laboratory Report

The FC:

- Reviews the *Case Details Report*, supporting data, and DEA-113 for accuracy.
- Signs and dates the DEA-113 electronically.
- Submits the case to the supervisor for technical and administrative review.
- Corrects any discrepancies identified by the reviewer.

The SC:

- Reviews the *Case Details Report*, the supporting data, and DEA-113 for technical and clerical accuracy, ensuring that:
  - Case, exhibit, and LIMS identifiers are properly documented.
  - Gross weight and evidence descriptions are complete and consistent with the DEA-7 or equivalent.
  - Observations and analyses are clearly and completely documented.
  - Analytical techniques are appropriate for the sample type.
  - Instrumental data and attachments are included and appropriately annotated (e.g., spectra, chromatograms, bulk photos).
  - Conclusions are supported by test results.
  - Manual calculations are accurate.

- All documents are free of administrative or transfer errors and improper use of abbreviations.
- Communicates and records any discrepancies or corrections to the analyst and returns the case electronically to the analyst for resolution.
  - For paper case files, discrepancies or corrections must be documented and included in the case file.
- Approves by signing and dating the DEA-113 electronically, thus signifying the following:

“After evaluating all reviewable data submitted with the *Case Details Report*, the reviewer agrees with the conclusions, to include the identification of the controlled substance(s) or other drugs, as reported by the analyst.”
- Submits the approved report for distribution in accordance with LOM 73.

#### 11.7 Analyzing and Reporting Foreign Drug Samples (SFL1 only)

SFL1:

- Analyzes enforcement REDACTED samples received from foreign offices.
- Reports and distributes the analytical results in accordance LOM 73.
  - For enforcement-only analysis, distribute a DEA-113 to the case agent in accordance with LOM 73.

#### 11.8 Revising Laboratory Reports (DEA-113)

The FC:

- Generates a supplemental DEA-113 when additional results become available or reanalysis is performed.
  - Select the *Final Report* option for the *Report Type*.
  - Includes the appropriate statement in the *Remarks* of the *Observations, Results, and Conclusions* section of the DEA-113 (See 2-9 and 2-10):

“Supplemental report to reflect *[Insert Reason]*. Refer to original laboratory report dated mm/dd/yyyy.”
  - Use the *Approved By* date from the original report.
- Generates an amended DEA-113 when errors on the original report are corrected.
  - Select the *Final Report* option for the *Report Type*.

- Includes the appropriate statement in the *Remarks of the Observations, Results, and Conclusions* section of the DEA-113:  
  
“Amended report to reflect *[Insert Reason]*. Refer to original laboratory report dated mm/dd/yyyy.”
- Use the *Approved By* date from the original report.

## CHAPTER 3 – FIELD ASSISTANCE

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## **1.0 Field Assistance**

### **1.1 Scope**

- Forensic support for field assistance can range from support of clandestine laboratory investigations to trace evidence collection, requiring vacuum sweeps and ion mobility spectrometry (IMS).
- Laboratory personnel use the procedures described in this chapter, REDACTED.

## **2.0 Clandestine Laboratories**

The Laboratory Director (LD) or designee:

- Coordinates all clandestine laboratory responses within the laboratory's area of responsibility in which DEA asserts primary authority.
- Ensures that all clandestine laboratory certified FCs have a working knowledge of the evidence processing procedures REDACTED.

### **2.1 Preparing for Clandestine Laboratory Investigations**

The FC:

- Briefs Special Agents (SAs) on technical matters pertinent to the investigation upon request.
- Ensures that the proper personal protective equipment (PPE) (e.g., respirators, goggles, etc.) will be at the site for use by all participating DEA laboratory personnel.
- Ensures that all participating FCs have a working knowledge of the methods of synthesis or manufacture for the drugs suspected of being produced in the laboratory under investigation.
- Ensures that all participating laboratory personnel are familiar with all information supplied to the field laboratory by the SA or Task Force Officer (TFO) regarding the investigation.

### **2.2 On Site Activities**

The FC:

- Enters the laboratory only after the premises are secured by SAs or TFOs.
- Conducts an assessment of the laboratory to identify potential hazards, the current state of the laboratory (e.g., dismantled, operational, in-process, etc.), and the sequence of synthetic steps used in the manufacturing process.
- Questions the suspected operating personnel, if necessary, to minimize a potentially hazardous situation regarding the current state of the laboratory and obtain information regarding possible safety concerns, synthesis routes, etc.
  - Specific authorization must be obtained from the senior, on-site SA or TFO prior to attempting any communication with REDACTED, operating personnel, or members of the press.
  - The SA or TFO must be present and document any communication with the REDACTED, operating personnel, or members of the press.
- Directs the shut-down of all operational equipment, if applicable, after determining the manufacturing sequence.



- For extraction laboratories involving butane, ensures the butane canister(s) has been turned off and is no longer off-gassing.
- Obtains approval from the SA or TFO prior to moving items that require relocation for safety reasons or to effectively assess the laboratory.

REDACTED

- Assists SAs or TFOs with debriefing suspected operating personnel involved in the investigation, and obtains information of a technical nature.
  - REDACTED
- Assists the SAs or TFOs in preparing a complete inventory of the laboratory, and in determining what items to seize as evidence, to include: tableting machines, punches, dies, glassware, etc.
- Photographs and/or records all essential areas of the clandestine laboratory, as well as exhibits seized. (See 2-2.5)
- Performs field tests on site, when applicable.
- Documents items seized as evidence with unique identifying information. Ensures that the items can be recognized in court.
- Assists SAs or TFOs in identifying solvents and other hazardous materials present at the laboratory site for proper disposal by hazardous waste contractors. REDACTED
- Assists SAs or TFOs with identifying chemicals, mixtures, and waste suspected of containing listed or controlled substances for on-site adulteration by the disposal company, REDACTED

## **2.3 Analysis and Reporting**

The LD or designee:

- When feasible, ensures that exhibits seized at a clandestine laboratory or processing site are assigned to a FC who participated in the operation.

The FC:

- Prepares a DEA-500, Clandestine Laboratory Report, when applicable, after all the exhibits from the clandestine laboratory have been analyzed.
- Reports production capabilities as 100% theoretical yields, based on amounts (either calculated or actual) of precursor material.
- Attaches a copy of the REDACTED, for DEA cases or similar available reports from other agencies to the original DEA-500.

- After completing the analysis of clandestine laboratory evidence in which there was not a participating DEA FC, prepares a DEA-500 upon request only.
- Retains all documentation, including (but not limited to): handwritten notes, hard copies of computer generated notes, photographs, sketches, or diagrams generated by laboratory personnel from an investigation outside of the laboratory in the case file.
- Offers expert opinions at trial regarding estimated actual yields, or upon receiving a written request from the prosecutor.

The SC:

- Reviews the DEA-500.
- Stamps all copies of the completed DEA-500 "*DEA Sensitive*."
- Attaches the DEA-500 in LIMS to *Case Attachments* as attachment type DEA-500.
- REDACTED
- Sends copies of the report to the following offices:
  - The office head or the designee of the office conducting the investigation
  - Special Agent in Charge (SAC) or Regional Director (RD) having line authority over the resident or district office, post of duty (POD), or country office (CO) conducting the investigation (if applicable)
  - REDACTED
  - Drug and Chemical Evaluation Section (DPE), Headquarters
  - Synthetic Drugs and Chemicals Section (DOS), Headquarters
- Forwards a copy of the transmittal letter(s) to the Office of Forensic Sciences.

### 2.3.1 Capacity Report Memorandum

The FC:

- After completing the analysis of extraction, recrystallization, tableting, or other laboratory evidence, prepares a Capacity Report Memorandum found on the SFDCC upon request only or when information not found in the DEA-500 is needed.
  - For laboratories in which there was not a participating DEA FC, measurements to prepare the memorandum are provided by the SA or TFO.

The SC:

- Reviews and approves the Capacity Report Memorandum.
- Attaches the Capacity Report Memorandum and any associated documentation REDACTED in LIMS to *Case Attachments* in LIMS.
- Sends the memorandum to the requesting SA or TFO
- Forwards a copy of the memorandum to SFL1.

### **3.0 Collecting Trace Drug Evidence**

The LD or designee:

- Assigns a FC to perform a trace evidence collection/vacuum search, upon request from a field office.
- Establishes the conditions and limitations of the trace evidence collection/vacuum search, in conjunction with requesting field office.

The FC:

- Accompanies the SAs, TFOs, or Diversion Investigators (DIs) to conduct a trace evidence collection/vacuum search for drug evidence.
- Discusses special evidence preservation precautions unique to trace evidence collection with the SAs, TFOs, or DIs, prior to entering any premises.

#### **3.1 Ion Mobility Spectrometer**

##### **3.1.1 Storing the IMS Equipment**

The FC:

- Stores all containers, including crates that store the supporting field supplies for the ion mobility spectrometer (IMS), in a clean, dry room.
- Does not expose any IMS equipment or travel supplies to moisture or controlled substances.

##### **3.1.2 Transporting the IMS Equipment**

The FC:

- Inspects and ensures that the IMS is operational, prior to deploying it for field operation.
- Transports the IMS in a travel crate.
- If the equipment is being shipped:
  - Labels the outside of the travel crates as “FRAGILE.”
  - Ensures a label is affixed to the IMS stating: “Contains a sealed radio-active source (Ni 63 at 15mCi),” if applicable. Categorization, labeling, and shipper’s declaration are not required.
- Locks the crate(s), if permitted by the shipping company or airline.
- Transports the IMS via air cargo, if permitted by airline regulations.

NOTE: The same declaration of radioactive materials is required.

- Hand-carries the computer.

### **3.1.3 Setting-Up and Inspecting Equipment On-Scene**

The FC:

- Inspects the equipment for any damage.
- Sets up the IMS, in accordance with the operator's manual.
- Avoids areas that could lead to potential contamination, exposure to water or moisture.
- Does not permit smoking near the instrument under any circumstances.
- Ensures that all items used during the analysis are free of contamination to include:
  - Filter
  - Filter cartridge
  - Remote sampling device
  - IMS instrument

### **3.1.4 Calibrating and Maintaining Equipment On-Scene**

The FC:

- Calibrates the IMS, in accordance with the operator's manual.
- Troubleshoots and, if possible, corrects any problems that occur in the field.

### **3.1.5 Collection Filters**

The FC:

- Follows laboratory procedures for the instrument when assembling the collection filters.

## **3.2 Collecting Evidence**

The FC:

- Collects samples as follows:
  1. Blank all filters to be used on the IMS.
    - If the blank is not clean, replace and re-blank a new filter or use the IMS repeatedly to burn (remove) the interfering material from the filter.

- Save plasmagrams as “blanks” on the computer hard drive with documentation that allows them to be linked to the collected samples.
- 2. Obtain an environmental blank with a blanked filter.
  - Vacuum the air near the IMS instrument, and check the sample on the IMS.
  - Save the plasmagram on the computer hard drive, so that the data can be linked back to the environmental blank.
  - Submit the environmental blank as an exhibit.
- 3. Using a previously blanked filter (step 1), collect samples for testing on a filter by a vacuum technique, or by a wiping technique.
- 4. Analyze the sample with the IMS, and save the corresponding results.
  - If there was a positive IMS result, vacuum the same area more thoroughly to obtain a “heavy” sample.
  - If the IMS was negative and a sample is not required for further analysis, save the plasmagram and include it with the case file. Document all negative results.
- 5. Photograph and/or record all essential areas of the sweep, as well as the exhibits seized (2-2.5).
- 6. Prepare a diagram of the area where the evidence was collected. Indicate where each exhibit was found or collected.
- Places collected samples in plastic sealed evidence envelopes (PSEE) as follows:
  - 1. Place each disk assembly in separate plastic bags and label appropriately.
  - 2. Place samples from each area swept and the corresponding environmental blank in separate PSEEs.
  - 3. Enter the case number and exhibit number provided by the SA, TFO, or DI on the PSEE.

NOTE: The environmental blank and the corresponding sample are given sequential Investigating Agency (IA) exhibit numbers (e.g., Exhibit 1 is the environmental blank and exhibit 2 is the corresponding heavy sample. Exhibit 3 will be the next environmental blank, and Exhibit 4 the heavy sample from the next area swept, etc.).

### **3.3 Submitting Evidence**

The FC:

- Turns the evidence over to the custody of the SA, TFO, or DI for submission to the laboratory.

### **3.4 Upon Return to Laboratory**

The FC:

- Restocks supplies and cleans the IMS.
- Notifies the instrument monitor of any instrument problems.
- Records any maintenance conducted in the field in the instrument maintenance logbook.

### **3.5 Reporting Results**

The FC:

- Reports the results on the DEA-113 after laboratory analysis. (See 2-5.0)

NOTE: A separate narrative report is not issued.

## **4.0 Processing Synthetic Drugs**

The LD or designee:

- Assigns a FC to accompany the SAs, TFOs, or DIs and to assess or sample a synthetic drug processing or storage facility.

The FC:

- Assists SAs, TFOs, or DIs with processing synthetic drug exhibits in the field following the guidelines outlined below:

### **4.1 Plant Material**

#### **4.1.1 Hazardous Materials**

- Sample 2 kg of material and submit as a single exhibit.
- Determine the weight of the bulk material.
- Photograph the bulk material.
- Turn the bulk material over to a hazardous waste contractor for adulteration and destruction.

#### **4.1.2 Non-Hazardous Materials**

- Sample 2 kg of material and submit it as a sub-exhibit of the bulk, designated with an “a” (e.g., sub-exhibit “1a”).
- Store the un-sampled bulk exhibit (e.g., exhibit “1”) at the laboratory or at the field division.

### **4.2 Powder Chemicals**

- Submit all material to the laboratory.

### **4.3 Retail Packages**

#### **4.3.1 Same Brand and Same Flavor**

- Under 2 kg of material inside packets:
  - Submit all packets as a single exhibit.
- Over 2 kg of material inside packets:
  - Sample 2 kg of material inside packets. Submit it as a sub-exhibit of the bulk, designated with an “a” (e.g., sub-exhibit “1a”). Use table in 4.5 as a guideline.



- Store the un-sampled bulk exhibit (e.g., exhibit “1”) at the laboratory or field division.

NOTE: In the event that different size packages are present, submit proportional sampling of each size package to meet total submission of 2 kg of material inside packets.

#### **4.3.2 Same Brand and Different Flavors**

- Under 2 kg of material inside packets:
  - Submit all packets.
- Over 2 kg of material inside packets:
  - Sample 2 kg of material inside packets that is representative of all flavors present as a sub-exhibit of the bulk designated with an “a” (e.g., sub-exhibit “1a”). Use table in 4.5 as a guideline.
  - Store the un-sampled bulk exhibit (e.g., exhibit “1”) at the laboratory or field division (OM waiver).

NOTE: In the event that different size packages are present, submit proportional sampling of each size package to meet total submission of 2 kg of material inside packets.

#### **4.3.3 Different Brands**

- Under 2 kg of material inside packets:
  - Submit all packets of each brand as separate exhibits (e.g., exhibits “1,” “2,” “3,” etc.).
- Over 2 kg of material inside packets:
  - Sample 2 kg of material inside packets in separate containers for each brand as sub-exhibits of the bulk exhibits designated with an “a” (e.g., sub-exhibits “1a,” “2a,” “3a,” etc.). Use table in 4.5 as a guideline.
  - Store the un-sampled bulk exhibits (e.g., “1,” “2,” “3,” etc.) at the laboratory or field division.

NOTE: In the event that different size packages are present, submit proportional sampling of each size package to meet total submission of 2 kg of material inside packets.

#### **4.4 Liquids**

- Do not submit commercially labeled solvent containers to the laboratory. Document and transfer them to a DEA hazardous waste contractor for processing.
- Solvents containing suspected controlled substances are sampled by the site safety officer or a clandestine laboratory certified FC.

- Estimate the total volume and submit 1 oz. to the laboratory. Adulterate the remaining liquid, prior to transport off-site.

#### 4.5 Sampling Table

Declared Weight on Packet	No. of Packets to Submit
0.5 g	4000
1 g	2000
3 g	667
5 g	400
10 g	200

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## 1.0 Fingerprint Program

### 1.1 Separating and Preserving DEA Fingerprint Evidence

The FC:

- Carefully separates all packaging from the alleged controlled drug substance, removing as much controlled drug substance as possible, leaving little or no residue.
- While conducting the separation, handles the physical evidence carefully to preserve any latent prints that might be present.
- Places the separated fingerprint evidence into an additional container (e.g., plastic bag).
  - Ensures that any drug paraphernalia is packaged to prevent accidental injury (e.g., covers exposed hypodermic needles, packages razor blades separately, etc.).
- Marks the additional container with the LIMS case number, initials, and date before inserting the separated fingerprint evidence.
- Places additional container into an evidence container.
- Seals and annotates the evidence container (e.g., Latent Print Examination).
- Identifies samples containing hazardous substances or objects (e.g., LSD, fentanyl analogues, drug paraphernalia or biological hazards). For example, “*Caution: Evidence contains [list specific hazard]*”, on the evidence container.
  - For evidence sent to another laboratory for latent print examination, includes a statement in the transmittal documents identifying the potential hazard.
- Completes the following lines on the evidence label:
  - CASE NUMBER – enter Investigating Agency (IA) case number
  - EXHIBIT NUMBER – enter laboratory exhibit number
  - SEALED BY – FC prints name, signs, and dates.
- Creates a Fingerprint Unit (FIN) in LIMS.
- Returns the fingerprint evidence to the vault.
- Includes the following statement in the “Remarks” of the *Exhibit Details* section of the DEA-113: “*Original packaging separated for latent print examination.*”

## 1.2 Preserving Fingerprint Evidence Not Separated from Drug Evidence

In some cases, the controlled drug substance present on material to be examined for latent fingerprints cannot be removed without destroying latent prints which may be present (e.g., LSD blotter papers).

The FC:

- Contacts the submitting agent to determine if both the drug analysis and the fingerprint processing are needed.
- Contacts a fingerprint specialist to determine the best way to handle and process the evidence if both examinations are needed.
- Annotates the evidence container according to 4-1.1.

## 1.3 Separating Fingerprint Evidence for Other Agencies

The FC:

- Reviews the submitted paperwork REDACTED for a fingerprint examination request.

NOTE: If it is impractical to separate other agency latent print/drug evidence, laboratory management will determine if and how to preserve the latent print evidence, or whether the latent print evidence will be examined at the DEA laboratory.

- Carefully separates all packaging from the alleged controlled drug substance as in 4-1.1.
- Creates additional units in LIMS.
  - Create Fingerprint Units (FIN) for containers being returned to the other agency for latent print examination. Route to CT.
  - Follow procedures in 4-1.1 for evidence being analyzed by DEA. Route to CT/LP with SC approval.
- Annotates the *Remarks* of the *Exhibit Details* section of the DEA-113: “*Original packaging separated and returned to [Insert Agency Name] for latent print examination.*”

## 1.4 Sampling for Fingerprint Examination of Bulk Drug Evidence Seizures

The Laboratory Director (LD) or designee:

- Determines fingerprint examination procedures for bulk seizures (i.e., the number of units to be examined), in consultation with the FC, Supervisory Fingerprint Specialist, Fingerprint Specialist (FS), and an appropriate enforcement official on a case-by-case basis.

## 1.5 Drug Evidence Packaging - Bench Transfers

The FC:

- Follows the bench transfer procedure below when the latent print examination will be conducted while the evidence is in the possession of the FC:
  - Add and complete the *Gross Weight* and *Description of Evidence* tests.  
  
NOTE: The FC may re-test the *Description of Evidence* after receiving the evidence from the FS.
  - Transfer the evidence to the FS in LIMS.
  - After fingerprint analysis and reassignment of the exhibit, receive the evidence from the FS in LIMS.
- Upon completion of the fingerprint and chemical analyses, reseals both the drug and original fingerprint evidence.
- Annotates the *Remarks* in the *Exhibit Details* section of the DEA-113: “*Original evidence submitted for latent print examination.*”

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Chapter 4 – Fingerprint REDACTED  
**Revision: 4**  
**Issue Date:** September 5, 2019  
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**Appendix 1A – Definitions**

Term	Definition
<b>Adulterant</b>	A non-controlled but pharmacologically active substance that may be added to a controlled substance.
<b>Accuracy</b>	The closeness of agreement between the experimental value and the true value.
<b>Analogue</b> (21 U.S.C. § 802)	<p>(A) Except as provided in subparagraph (B), the term "controlled substance analogue" means a substance:</p> <ul style="list-style-type: none"> <li>(i) the chemical structure of which is substantially similar to the chemical structure of a controlled substance in schedule I or II;</li> <li>(ii) which has a stimulant, depressant, or hallucinogenic effect on the central nervous system that is substantially similar to or greater than the stimulant, depressant, or hallucinogenic effect on the central nervous system of a controlled substance in schedule I or II; or</li> <li>(iii) with respect to a particular person, which such person represents or intends to have a stimulant, depressant, or hallucinogenic effect on the central nervous system that is substantially similar to or greater than the stimulant, depressant, or hallucinogenic effect on the central nervous system of a controlled substance in schedule I or II.</li> </ul> <p>(B) Such term does not include:</p> <ul style="list-style-type: none"> <li>(i) a controlled substance;</li> <li>(ii) any substance for which there is an approved new drug application;</li> <li>(iii) with respect to a particular person any substance, if an exemption is in effect for investigational use, for that person, under section 505 of the Federal Food, Drug, and Cosmetic Act (21 U.S.C. 355) to the extent conduct with respect to such substance is pursuant to such exemption; or</li> <li>(iv) any substance to the extent not intended for human consumption before such an exemption takes effect with respect to that substance.</li> </ul>

Term	Definition
<b>Analytical Scheme</b>	The combination of sampling protocols and tests forming the core of the DEA laboratory identification process. DEA's analytical scheme consists of presumptive and confirmatory analyses involving the use of methods developed and validated to be fit for the identification of controlled and non-controlled substances. An effective analytical scheme encompasses suitable tests that, when combined, address limitations that may preclude a conclusive identification.
<b>Bias</b>	The difference between the expectation of the test results and an accepted reference value.
<b>Biohazard</b>	Infectious agents or hazardous biological materials that present a risk or potential risk to the health of humans.
<b>Bulk exhibit</b>	Contraband drug evidence submitted to a DEA laboratory whose net weight exceeds the threshold amount.
<b>Bulk portion</b>	Amount of contraband drug evidence in excess of the appropriate threshold amount.
<b>Case Details Report (CDR)</b>	The summary of analytical testing within LIMS.
<b>Certified reference material (CRM)</b> <b>(ISO Guide 30:1992(E)/Amd.1:2008)</b>	Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.
<b>Co-analysis</b>	Comparison of an analyte and reference material in the same experiment by simultaneous analysis.
<b>Combined standard uncertainty (<math>u</math>)</b>	Standard measurement uncertainty that is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model. In case of correlations of input quantities in a measurement model, covariances must also be taken into account when calculating the combined standard measurement uncertainty.
<b>Composite</b>	Representative, homogenized material prepared in accordance with the Evidence Sampling Plan (ADM 2-4) and the sampling procedures in Appendix 2C.
<b>Confirmation technique</b>	Analytical test that provides distinctive structural information to identify a substance. The test must be appropriate for the sample and analyte and may include the following: IR, MS, Raman, or NMR.

Term	Definition
<b>Confirmed</b>	See Reporting Terms
<b>Correlated measurements</b>	Measurements that are not independent of each other or that are dependent on a common third quantity. The uncertainty associated with the combination of correlated uncertainties is obtained by the linear sum of the individual uncertainties.
<b>Coverage factor</b>	Number larger than one by which a combined standard measurement uncertainty is multiplied to obtain an expanded measurement uncertainty. A coverage factor is usually symbolized as <i>k</i> .
<b>Critical resolution pair</b>	For separation analyses, a pair of compounds eluting or migrating with a baseline resolution between 1.5 and 5.0 at half-height.
<b>Determined</b>	Use of one test to obtain information (e.g., salt form, purity, isomer). For use on DEA-113, see Reporting Terms.
<b>Diluent</b>	A substance typically used to increase the bulk of a finished product.
<b>Exhibit</b>	Physical evidence submitted to the laboratory. See also Sub-exhibit.
<b>Expanded uncertainty (U)</b>	Product of a combined standard measurement uncertainty and a coverage factor. The coverage factor depends upon the type of probability distribution of the output quantity in a measurement model and on the selected coverage probability.
<b>Gummy exhibit</b>	Exhibit which is not amenable to grinding or mixing.
<b>Identified</b>	See Reporting Terms.
<b>Increment</b>	Randomly chosen portion from the exhibit from which the composite is assembled.
<b>Instrument blank</b>	A quality control measure, run immediately prior to the sample of interest, to ensure an instrument is free of contamination and suitable to utilize for analysis.
<b>Investigating Agency (IA)</b>	The law enforcement agency submitting the physical evidence to the laboratory. The case number and exhibit number from the DEA-7 are entered into LIMS as the IA case number and IA exhibit number, respectively.
<b>Laboratory exhibit number</b>	A LIMS specific field, which directly relates to the IA exhibit number, used to designate laboratory created sub-exhibits.

Term	Definition
<b>LIMS</b>	Laboratory information management system
<b>LIMS case file</b>	Electronic record of all actions performed on a piece of evidence. The record may include the items found in LOM 73.
<b>LIMS case number</b>	A unique identifier which refers to a single IA exhibit.
<b>Linearity</b>	The ability of a method to produce test results that are directly proportional to analyte concentration within a given range.
<b>Low-response compound</b>	Compound that produces a low-intensity signal under routine experimental conditions.
<b>Measurand (VIM, 3<sup>rd</sup> Ed.)</b>	Quantity intended to be measured. <ul style="list-style-type: none"> <li>• Net weight - The measured weight of an exhibit.</li> <li>• Purity - The measured fraction of an exhibit associated with the identified substance.</li> <li>• Amount of Pure Drug - The calculated amount of actual identified substance in an exhibit defined by the net weight multiplied by the purity.</li> </ul>
<b>Measurement assurance (ASCLD/LAB- International AL-PD- 3059)</b>	Practices put into place to monitor a testing or calibration process and to ensure the calibration status of equipment, reference standards, or reference materials used in a measurement process.
<b>Measurement uncertainty</b>	Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand.
<b>Method</b>	The combination of the technique (separation, confirmation, or hyphenated), the specific instrument used, and all associated operating parameters required for an analysis.
<b>Method validation</b>	The process by which it is established, through laboratory studies, that the performance characteristics of a procedure meet the requirements for the intended analytical applications.
<b>Negative control (blank)</b>	A quality control measure to verify that the reagents, analysis protocols, and instruments are free of contamination and neither interferes with the results, nor affects the analytical signal.

Term	Definition
<b>Orthogonal techniques</b> (ULTR for General Forensic Chemistry and Seized Drug Examinations)	Two or more techniques that utilize different fundamental principles of selectivity for characterizing an analyte or class of analytes.
<b>Physical evidence</b>	May consist of drugs, chemicals, laboratory equipment, packaging, photographs, documents, latent prints, digital devices or media, money, or any other tangible items and may be used to establish a violation of law.
<b>Positive control</b>	Qualitative Methods: A verified reference material used as a quality control measure to demonstrate that the analyte of interest is detected and produces the expected result.  Quantitative Methods: A quality control (QC) sample used to demonstrate that the analyte of interest is detected and produces the expected purity.
<b>Presumptive test</b>	Analytical test that provides an indication of the sample composition. The test must be appropriate for the sample and analyte and may include: chemical tests, color tests, microcrystalline tests, optical crystallography, UV-Vis spectrophotometry, and separation techniques.
<b>Probe technique</b>	A procedure whereby units are pierced or small openings are made and a small amount of material is removed for analysis.
<b>Procedural blank</b>	A quality control measure used to verify that reagents, solvents, and labware are free of contamination and evaluated immediately prior to the sample analysis. Consists of the matrix (to include, but not limited to, the solvent for a separation technique, or KBr for IR) which has been taken through every step of the analytical protocol using the same glassware, reagents, solvents, and analytical instrument.
<b>Quality control (QC) sample</b>	A material that is well characterized in-house, or by a third party, which contains known amounts of analyte(s). The composition of a QC sample should mimic routine compositions received by the laboratory.
<b>Quality control (QC) solution</b>	Testing solution prepared by diluting a known amount of a QC sample in a known volume of appropriate solvent, based on the method being tested.
<b>Quality control (QC) check</b>	Quantitative analysis of two QC solutions during quantitative method validation containing the target analyte at a concentration representing the high and low end of the method working range, used to verify the method's accuracy and repeatability.

Term	Definition
<b>Reagent</b>	Substance used in a reaction for the purpose of detecting, measuring, examining, or analyzing other substances.
<b>Reference compound</b>	A component in a performance mixture or an internal standard (e.g., tetracosane, resorcinol, etc.) that is used for the purpose of area/height/time comparisons.
<b>Reference material (RM)</b>	A homogeneous, stable, and traceable material which has been established to be fit for use in identification or quantitation of substances.
<b>Relative area</b>	The ratio of the peak area of one compound relative to the peak area of a reference compound.
<b>Relative retention / migration time</b>	The ratio of the elution (retention/migration time) of one compound relative to the elution (retention/migration time) of a reference compound.
<b>Relative standard deviation (RSD)</b>	For replicate measurements, the measured standard deviation divided by the mean.
<b>Repeatability</b>	The degree of agreement among individual test results when the procedure is applied multiple times, over a short time interval.
<b>Reporting terms:</b>	Used for reporting results that fulfill the minimum requirements of the DEA analytical scheme (e.g., 2 portions-2 tests for controlled substances, 1 portion-2 tests for adulterants).
<b>Identified or Confirmed</b>	
<b>Determined</b>	Used for reporting results for salt form, optical isomer, and purity.
<b>Residue</b>	A quantity of substance for which the determination of a weight is not practical or the weight is less than 10 mg.
<b>Reviewable data</b>	Information obtained from analytical methodology or documents containing recorded forensic observations.
<b>Ruggedness</b>	The ability of a measurement process to withstand small uncontrolled or unintentional changes in its operating conditions. Ruggedness is a measure of reproducibility of test results under the variation in conditions normally expected from laboratory to laboratory and from analyst to analyst.
<b>Selectivity</b>	The separation of the analyte(s) of interest and the internal standard, if utilized, from other sample components in a mixture/matrix.

Term	Definition
<b>Selectivity solution</b>	Method-specified solution containing the target analyte and additional components at concentrations commonly encountered in laboratory submissions.
<b>Separation technique</b>	Analytical test used to evaluate possible multi-component mixtures. The test must be appropriate for the sample and analyte and may include: TLC, GC, LC, CE, soft ionization MS, and IMS. Separation may be based on time or mass.
<b>Standard uncertainty</b>	Measurement uncertainty expressed as a standard deviation.
<b>Sub-exhibit</b>	The separation of an exhibit resulting from significantly different chemical composition, color, appearance, etc.
<b>Target analyte(s)</b>	Substance(s) to be identified (qualitative analysis) or measured (quantitative analysis). For qualitative analysis, it is the common analyte(s) that is identified in all selected units. For quantitative analysis, it is the analyte(s) for which purity is determined.
<b>Technique</b>	Wet chemical or instrumental tests that provide information about the composition of a substance. Examples include mass spectrometry, infrared spectroscopy, or color tests.
<b>Test portion</b>	The amount withdrawn for qualitative or quantitative analysis.
<b>Threshold amount</b>	<p>The required size of a representative sample from a bulk exhibit involving the following Schedule I and II Controlled Substances:</p> <ul style="list-style-type: none"> <li>• <b>Heroin:</b> 2 kg of a mixture or substance containing a detectable amount of heroin.</li> <li>• <b>Cocaine:</b> 10 kg of a mixture or substance containing a detectable amount of: <ul style="list-style-type: none"> <li>○ Coca leaves, except coca leaves and extracts of coca leaves from which cocaine, ecgonine, and derivatives of ecgonine or their salts have been removed.</li> <li>○ Cocaine, its salts, optical and geometric isomers, and salts of isomers.</li> <li>○ Ecgonine, its derivatives, their salts, isomers, and salts of isomers.</li> <li>○ Any compound, mixture, or preparation which contains any quantity of any of the substances referred to in the preceding three bullet points.</li> </ul> </li> <li>• <b>Cocaine base:</b> 10 kg of a mixture or substance containing cocaine base.</li> </ul>

(Continued below)



Term	Definition
<b>Threshold amount (Continued)</b>	<ul style="list-style-type: none"> <li>• <b>PCP:</b> 200 g of powdered phencyclidine (PCP) or two kilograms of a powdered mixture or substance containing a detectable amount of phencyclidine (PCP) or 28.35 g of a liquid containing a detectable amount of phencyclidine (PCP).</li> <li>• <b>LSD:</b> 20 g of a mixture or substance containing a detectable amount of Lysergic Acid Diethylamide (LSD).</li> <li>• <b>Fentanyl:</b> 800 g of a mixture or substance containing a detectable amount of N-phenyl-N-[1-(2-phenylethyl)-4-piperidinyl] propanamide [commonly known as fentanyl].</li> <li>• <b>Fentanyl analogues:</b> 200 g of a mixture or substance containing a detectable amount of any analogue of N-phenyl-N-[1-(2-phenylethyl)-4-piperidinyl] propanamide. Reflective of 28 CFR 50.21 with corrected nomenclature from <i>The Merck Index</i>.</li> <li>• <b>Hashish:</b> 20 kg of hashish or two kilograms of hashish oil [21 USC 841(b)(1)(D), 960(b)(4)].</li> <li>• <b>Other Schedule I or II:</b> 2 kg of a mixture or substance containing a detectable amount of any Schedule I or II contraband substance in the Controlled Substances Act for which no specific threshold amount has been specified above.</li> <li>• <b>Marijuana:</b> 10 kg of a mixture or substance containing a detectable amount of marijuana.</li> <li>• In the event of any changes to Section 401(b)(1) of the Controlled Substances Act [21 USC 841(b)(1)] as amended occurring after the date of these regulations, the threshold amount of any substance therein listed, except marijuana, shall be twice the minimum amount requested for the most severe mandatory minimum sentence.</li> </ul>
<b>Traceability (VIM, 3<sup>rd</sup> Ed., 2.41)</b>	<p>The property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty.</p>

Term	Definition
<b>Unique identifier</b>	<p>For the purposes of documenting, analyzing, and preserving physical evidence within the laboratory, each exhibit's unique identifier is the LIMS case number. However, for sub-exhibits, the unique identifier shall consist of the LIMS case number and the sub-exhibit number.</p> <p>For weighing events, the unique identifier is either the container ID, the universal weight ID, or the test ID barcode.</p> <p>For reference materials, the identification number that provides traceability.</p>
<b>Uncorrelated measurements</b>	<p>Independent measurements subject only to random sources of uncertainties. The uncertainty associated with the combination of uncorrelated measurements is obtained by the quadratic sum of the individual uncertainties.</p>
<b>Working range</b>	<p>The inclusive interval between the upper and lower levels of analyte concentration that have been demonstrated to fulfill the acceptance criteria required for repeatability, accuracy, and linearity for the validation of a given method. During analysis, the working range is further limited by the analyte concentration of the QC solutions used and must not exceed the upper and lower ends of the linear range.</p>

## Appendix 1B – Acronyms and Abbreviations

- Listed below are the acronyms and abbreviations for use by DEA laboratory personnel. The acronyms and abbreviations are not case sensitive.
- Acronyms and abbreviations defined in the following references are also approved for use.
  - American Chemical Society (ACS) Style Guide
  - Chemical Abstracts Services (CAS) Standard Abbreviations and Acronyms Listing B
  - Diversion Control Division - Controlled Substance Schedules
  - Laboratory Operations Manual (LOM)
  - Merck Index
  - Official Methods of Analysis of AOAC International
  - SWGDRUG Drug Monographs
- Abbreviations for the latent print program can be found in Appendix 1B of the Latent Print Examination Manual (LPEM), while abbreviations for the digital evidence program can be found in Appendix B of the REDACTED.
- Any abbreviation that is to be pluralized must be followed with the letter “s” (example: PBs, PKGs, ZPBs). The addition of a letter “s” does not constitute a new abbreviation.
- Any abbreviations not listed below or in the above references must be defined in the case details report upon first use.

Compounds	
<b>6MAM</b>	O6-Monoacetylmorphine
<b>AC</b>	Acetylcodeine
<b>AMPH</b>	Amphetamine
<b>APAP</b>	Acetaminophen
<b>BICARB</b>	Bicarbonate
<b>BMPEA</b>	$\alpha$ -benzyl-N-methyl- $\beta$ -phenethylamine
<b>C13</b>	Tridecane
<b>C20</b>	Eicosane
<b>C22</b>	Docosane
<b>C24</b>	Tetracosane
<b>CAFF</b>	Caffeine
<b>CBC</b>	Cannabichromene
<b>CBD</b>	Cannabidiol
<b>CBG</b>	Cannabigerol

Compounds	
<b>CBN</b>	Cannabinol
<b>COC</b>	Cocaine
<b>DMSO2</b>	Dimethyl Sulfone
<b>DMTP</b>	Dimethylterephthalate
<b>EPHED</b>	Ephedrine
<b>HER</b>	Heroin
<b>METH</b>	Methamphetamine
<b>MJ</b>	Marijuana
<b>NN-DMA</b>	N,N-Dimethylamphetamine
<b>OXY</b>	Oxycodone
<b>PDMAB</b>	para-Dimethylaminobenzaldehyde
<b>PDMAC</b>	para-Dimethylaminocinnamaldehyde
<b>PSEUDO</b>	Pseudoephedrine
<b>PTHIT</b>	Phenyltetrahydroimidazothiazole
<b>8-THC</b>	Delta-8-Tetrahydrocannabinol
<b>THC</b>	Delta-9-Tetrahydrocannabinol

Evidence Descriptions	
<b>BC</b>	Body Carry
<b>Cap</b>	Capsule
<b>CKB</b>	Compressed Kilo Brick
<b>CM</b>	Crystalline Material
<b>KB</b>	Kilo Brick
<b>Plt Mat</b>	Plant Material
<b>Tab</b>	Tablet

Instrumentation	
<b>APCI</b>	Atmospheric Pressure Chemical Ionization
<b>CE</b>	Capillary Electrophoresis
<b>DART</b>	Direct Analysis in Real Time
<b>DESI</b>	Desorption Electrospray Ionization
<b>IMS</b>	Ion Mobility Spectrometry
<b>LTM</b>	Low Thermal Mass

Instrumentation	
<b>QTOF</b>	Quadrupole Time of Flight
<b>UPLC</b>	Ultra Performance Liquid Chromatography

LIMS	
<b>CDR</b>	Case Details Report
<b>CIF</b>	Combined Instrument Files
<b>CR</b>	Central Receiving
<b>CT</b>	Chemical Testing
<b>IA</b>	Investigative Agency
<b>LASS</b>	Laboratory Activity Summary Sheet
<b>SDMS</b>	Scientific Data Management System
<b>SO</b>	Storage Only
<b>REDACTED</b>	REDACTED

Miscellaneous	
<b>Alkd</b>	Alkaloid
<b>ATM</b>	Adulterant Test Mixture
<b>B/C</b>	Because
<b>Bioh</b>	Biohazard
<b>Bkgd</b>	Background
<b>BI</b>	Blank
<b>Clan Lab</b>	Clandestine Laboratory
<b>Comp</b>	Composite
<b>REDACTED</b>	REDACTED
<b>Decon</b>	Decontamination
<b>DNMC</b>	Does Not Meet Criteria
<b>Eff</b>	Effervescence
<b>ESP</b>	Evidence Sampling Plan
<b>REDACTED</b>	REDACTED
<b>Ex</b>	Exhibit
<b>GWAA</b>	Gross Weight After Analysis
<b>GW</b>	Gross Weight
<b>Haz</b>	Hazardous
<b>HQC</b>	High Quality Control Sample

<b>Miscellaneous</b>	
<b>REDACTED</b>	REDACTED
<b>Id</b>	Identification
<b>Inconcl</b>	Inconclusive
<b>Ind</b>	Indicated
<b>INO</b>	Insolubles Not Observed
<b>IO</b>	Insolubles Observed
<b>IS</b>	Internal Standard
<b>ISS</b>	Internal Standard Solution
<b>LQC</b>	Low Quality Control Sample
<b>Macro</b>	Macroscopic exam
<b>Micro</b>	Microscopic exam
<b>Mod</b>	Modified
<b>REDACTED</b>	REDACTED
<b>MT</b>	Migration Time
<b>NCS</b>	No Controlled Substance
<b>NCSD</b>	No Controlled Substance Detected
<b>NFA</b>	No Further Action
<b>NW</b>	Net Weight
<b>Orig</b>	Original
<b>PBI</b>	Procedural Blank
<b>QNS</b>	Quantity Not Sufficient
<b>QS</b>	Quantity Sufficient
<b>Qual</b>	Qualitative
<b>Quant</b>	Quantitative
<b>Repst</b>	Representative
<b>Rgt</b>	Reagent
<b>RM</b>	Reference Material
<b>RMT</b>	Relative Migration Time
<b>RT</b>	Retention Time
<b>RTV</b>	Return to Vault
<b>RW</b>	Reserve Weight
<b>Rxn</b>	Reaction
<b>SC</b>	Supervisory Chemist
<b>Scrn</b>	Screen
<b>SFC</b>	Senior Forensic Chemist

Miscellaneous	
<b>Sol</b>	Soluble
<b>Spl</b>	Sample
<b>SU</b>	Salt Form Undetermined
<b>TW</b>	Tare Weight
<b>Und</b>	Undetermined
<b>Unk</b>	Unknown
<b>W/</b>	With
<b>W/O</b>	Without
<b>Subst</b>	Substitute

Packaging	
<b>BT</b>	Black Tape
<b>CB</b>	Carbon Paper
<b>Cello</b>	Cellophane
<b>CKPB</b>	Clear Knotted Plastic Bag
<b>CP</b>	Clear Plastic
<b>CPB</b>	Clear Plastic Bag
<b>CPW</b>	Clear Plastic Wrap
<b>CZPB</b>	Clear Zip-Lock Plastic Bag
<b>EE</b>	Evidence Envelope
<b>Env</b>	Envelope
<b>GB</b>	Glassine Bag
<b>GT</b>	Grey Tape
<b>HS</b>	Heat Sealed
<b>KPB</b>	Knotted Plastic Bag
<b>NZPB</b>	New Ziplock Plastic Bag
<b>OZPB</b>	Original Ziplock Plastic bag
<b>PB</b>	Plastic Bag
<b>PKG</b>	Package
<b>PSB</b>	Plastic Sandwich Bag
<b>PSEE</b>	Plastic Sealed Evidence Envelope
<b>PW</b>	Plastic Wrap
<b>SSEE</b>	Self-Sealing Evidence Envelope
<b>TT</b>	Tan Tape

Packaging	
<b>VSB</b>	Vacuum Seal Bag
<b>WPB</b>	Whirl Pack Bag
<b>ZPB</b>	Ziplock Plastic Bag

Reagents	
<b>BSA</b>	N,O-Bis(Trimethylsilyl) Acetamide
<b>BSTFA</b>	N,O-Bis(Trimethylsilyl) Trifluoroacetamide
<b>CoSCN</b>	Cobalt Thiocyanate
<b>DIPC</b>	Diisopropylcarbodiimide
<b>DuqL</b>	Duquenois-Levine
<b>MRQ</b>	Marquis Reagent
<b>M Scott</b>	Modified Scott's Reagent
<b>MSTFA</b>	N-Methyl-N-(Trimethylsilyl) Trifluoroacetamide
<b>MTPA</b>	alpha-Methoxy-Alpha-Trifluoromethylphenylacetic Acid
<b>MTPA-Cl</b>	alpha-Methoxy-Alpha-Trifluoromethylphenylacetyl Chloride
<b>NaNP</b>	Sodium Nitroprusside
<b>PIT</b>	Phenylisothiocyanate
<b>TDTA</b>	di-p-Toluoyl-(d/l) Tartaric Acid
<b>TFAA</b>	Trifluoroacetic Acid
<b>TPC</b>	N-Trifluoroacetyl-L-Prolyl-Chloride

Solvents	
<b>SolvA-0.01</b>	0.01 mg/mL C24 in 4:1 Chloroform: Methanol
<b>SolvA-0.0125</b>	0.0125 mg/mL C24 in 4:1 Chloroform: Methanol
<b>SolvA-0.05</b>	0.05 mg/mL C24 in 4:1 Chloroform: Methanol
<b>SolvB-0.01</b>	0.01 mg/mL C24 in Ammonia-Saturated Chloroform
<b>SolvB-0.0125</b>	0.0125 mg/mL C24 in Ammonia-Saturated Chloroform
<b>SolvC-0.0125</b>	0.0125 mg/mL C24 in Chloroform with 10% Sodium Hydroxide
<b>SolvD</b>	0.4 mg/mL C24 in 9:1 Chloroform: Methanol
<b>THC-ISS</b>	0.05 mg/mL 4-androsten-3,17-dione in 9:1 MeOH:Chloroform
<b>β-CD</b>	beta-Cyclodextrin
<b>ACN</b>	Acetonitrile
<b>AF 3.7</b>	10mM Ammonium Formate Buffer, pH 3.7
<b>AMH</b>	Ammoniacal Hexane



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Solvents	
AMC	Ammoniacal Chloroform
DI	Deionized Water
Et2O	Diethyl Ether
EtOH	Ethanol, Ethyl Alcohol
Hex	Hexane
IPA	Isopropyl Alcohol
MeCl2	Methylene Chloride
MeOH	Methanol
Pet Ether	Petroleum Ether

## Appendix 1C – Instrument Maintenance Schedule

### 1 Capillary Electrophoresis

Frequency of Check	Parameter	Procedure
<b>Date of operation</b>	Cleanliness	Inspect buffer reservoirs/vials for potential microbial growth and determine if the buffer needs to be replaced. Inspect and fill reservoirs and check electrodes.
<b>Monthly</b>	Detector	Perform Diode Array Detector test. Replace the lamp(s) when necessary.
<b>Every 3 months</b>	Pressure system	Examine the inlet and outlet seals. Replace the air filter if applicable.

### 2 Liquid Chromatograph

Frequency of Check	Parameter	Procedure
<b>Monthly</b>	Detector	Using the column eluent, perform a diode array detector intensity check for max and min levels and perform a wavelength calibration.
<b>Every 2 years</b>	Cleanliness	Perform a check of the system. Check and clean or replace solvent inlet filters, buffer reservoirs, as needed.
<b>Every 2 years or if system performance deteriorates</b>	System parts	Take down instrument and examine the system. Change pre-column, column, lamp(s), replace pump head seals, pistons, and check valves as needed.

### 3 Gas Chromatograph

Frequency of Check	Parameter	Procedure
Monthly	Septum	Replace septum. Refer to manufacturer's recommendations for longer lasting septa.
Every 3 months	Split liner	Take down instrument and examine. Replace the split liner.
Yearly	Split line	Remove tubing and inspect. Clean if necessary.
Yearly or if system performance deteriorates	Gold seal and syringe	Take down instrument and examine. Replace the gold seal and syringe if necessary.
Yearly	FID	Clean and/or replace if necessary.

### 4 Fourier Transform Infrared Spectrophotometer (with or without ATR attachment)

Frequency of Check	Parameter	Procedure
Monthly	Cleanliness	Ensure area is free of possible contaminants.

### 5 Fourier Transform Infrared Spectrophotometer with Gas Chromatograph

Frequency of Check	Parameter	Procedure
Monthly	Cleanliness	Ensure area is free of possible contaminants.

### 6 Fourier Transform Raman Spectrophotometer

Frequency of Check	Parameter	Procedure
Monthly	Cleanliness	Ensure area is free of possible contaminants.

## 7 Mass Spectrometer with Gas Chromatograph

Frequency of Check	Parameter	Procedure
Yearly or if system performance deteriorates	Source	Clean the source. Replace if necessary.
Every 6 months	Pump	Check pump oil if applicable. Replace and/or fill as necessary.

## 8 Mass Spectrometer with Liquid Chromatograph

Frequency of Check	Parameter	Procedure
Every 3 months or if system performance deteriorates	Source (Electrospray)	Clean spray chamber and capillary spray shield. Replace if necessary.
Every 3 months or if system performance deteriorates	Source (APCI)	Clean corona needle if in use. Replace after 3 months of use or earlier if necessary.
Every 6 months or if system performance deteriorates	Capillary Spray Shield	Abrasively clean the spray shield. Replace if necessary.
Every 6 months	Pump	Change rough pump oil if applicable. Replace and/or fill as necessary.
If system performance deteriorates	Gas Conditioner	Replace.
Yearly or if system performance deteriorates	Source	Check nebulizer needle if in use. Replace if necessary.
If system performance deteriorates	Detector	Replace Electron Multiplier horn.

## 9 High Resolution Nuclear Magnetic Resonance Spectrometer

Frequency of Check	Parameter	Procedure
Weekly	Liquid nitrogen	Fill to capacity.
Every 4 months or if it drops below 50% full	Liquid helium	Fill to capacity.

## 10 Inductively-Coupled Plasma Mass Spectrometer

Frequency of Check	Parameter	Procedure
Monthly	Sample and peristaltic pump tubing	Check and replace if necessary
Every 3 months	Rotary Pump	Check oil level and color.
Yearly	Nebulizer	Clean and replace if necessary.
Yearly	Cooling Water Filter	Check.
Yearly	Sampling Cone/ Skimmer Cone	Clean and replace if necessary.
Yearly	Rotary Pump	Replace oil.
Yearly	Lenses	Clean and replace if necessary.
Yearly	Torch	Clean and replace if necessary.
If system performance deteriorates	Plasma Gas Tubing	Inspect for leaks.
If system performance deteriorates	Carrier Gas Tubing	Inspect for leaks.
Every 2 years	Oil Mist Filter of Rotary Pump	Inspect for leaks.
If system performance deteriorates	Electron Multiplier	Evaluate the electron multiplier. Replace if necessary.

**11 Elemental Analysis Isotope Ratio Mass Spectrometer**

Frequency of Check	Parameter	Procedure
Yearly	Source	Check the source. Clean or replace as necessary.
Yearly	Pumps	Change vacuum pump oil.

**12 Polarimeter**

Frequency of Check	Parameter	Procedure
Yearly	Cleanliness	Ensure area is free of possible contaminants.

**13 Microscopes**

Frequency of Check	Parameter	Procedure
Yearly	Cleanliness	Ensure area is free of possible contaminants.

**14 Balances/Microbalances**

Frequency of Check	Parameter	Procedure
Yearly	Cleanliness	Ensure area is free of possible contaminants.

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## 15 Ion Mobility Spectrometer

Frequency of Check	Parameter	Procedure
Monthly	Gasket, Inlet door, O-rings	Inspect and clean. Replace if necessary.
Monthly	Detector Inlet	Inspect and clean. Replace if necessary.
Monthly	Inlet liner	Inspect and clean. Replace if necessary.
Every 6 months	Source	Perform radiation leak test (National Leak Test Center of Nuclear Regulatory Commission).
Monthly	Condenser	Visually inspect and clean. Change material when one-half is discolored.
Date of Operation or monthly	Air purification unit	Check for visual discoloration. Change desiccant and charcoal in necessary.

## Appendix 1D – Color Test Reagent Preparation and Procedures

Results listed below are not comprehensive and are subject to the amount and purity of material tested. Additional results and color tests can be found in the references listed in ADM 1-11.

4-AP Test	
<b>Reagent</b>	A. Dissolve 300 mg 4-aminophenol in 995 mL ethanol and 5 mL 2N HCl. Store in amber glass bottle; refrigerate.  B. Dissolve 30 g NaOH in 300 mL water and 700 mL ethanol. Store in amber glass bottle; refrigerate.
<b>Procedure</b>	Place approximately 5 mg of sample in a test tube or well plate. Cover the sample with solution A. Add 2-4 drops of solution B. Observe color change.
<b>Results</b>	<i>THC:CBD &gt;1</i> Blue
<b>Results</b>	<i>THC:CBD &lt;1</i> Pink
<b>Results</b>	<i>Cannabinol</i> Blue
<b>Results</b>	<i>Δ<sup>8</sup>-THC</i> Blue
<b>Results</b>	<i>Cannabidiol</i> Pink
<b>Results</b>	<i>Cannabigerol</i> Pink
<b>Results</b>	<i>Cannabichromene</i> No color change
<b>Results</b>	<i>Cannabicyclol</i> Blue
<b>Results</b>	<i>Basil (dried)</i> No color change
<b>Results</b>	<i>Oregano (dried)</i> Green to Blue
<b>Results</b>	<i>Parsley (dried)</i> No color change
<b>Results</b>	<i>Sage (dried)</i> Green to Blue



Aldehyde-Oxidation H <sub>2</sub> SO <sub>4</sub> Reagent C-2	
<b>Reagent</b>	Add 1 drop of 37% formaldehyde solution to 5 mL concentrated sulfuric acid. Mix with 0.2 mL of 10% aqueous ferric sulfate solution.
<b>Results</b>	<i>Oxycodone</i> Blue

Chen's	
<b>Reagent</b>	A. 1% acetic acid solution B. 1% copper sulfate solution C. 2 N NaOH NOTE: A negative control should be done alongside samples for comparison due to similarity of color between reagent and positive result.
<b>Results</b>	<i>Pseudoephedrine</i> Purple
<b>Results</b>	<i>Ephedrine</i> Purple
<b>Results</b>	<i>Phenylpropanolamine</i> Purple
<b>Results</b>	<i>Norpseudoephedrine</i> Purple

Cobalt (II) Thiocyanate, Acidified	
<b>Reagent</b>	Dissolve 2.0 g cobalt (II) thiocyanate in 99 mL distilled water and 1 mL concentrated hydrochloric acid.
<b>Results</b>	<i>Cocaine</i> Blue flaky precipitate
<b>Results</b>	<i>Methaqualone</i> Blue
<b>Results</b>	<i>Phenyltetrahydroimidazothiazole</i> Blue

<b>Cobalt (II) Thiocyanate, Unacidified</b>	
<b>Reagent</b>	Dissolve 2.0 g cobalt (II) thiocyanate in 100 mL distilled water.
<b>Results</b>	<i>Cocaine Hydrochloride</i> Blue
<b>Results</b>	<i>Phenyltetrahydroimidazothiazole HCl</i> Blue

<b>Cobalt (II) Thiocyanate with Stannous Chloride</b>	
<b>Reagent</b>	A. Dissolve 2.0 g cobalt (II) thiocyanate in 100 mL distilled water. B. 5.0 g of stannous chloride in 90 mL distilled water and 10 mL concentrated hydrochloric acid.
<b>Procedure</b>	Place a few drops of solution A into the well of a spot plate. Add sample. If a blue precipitate develops in the solution, add solution B and observe whether or not the precipitate disappears.
<b>Results</b>	<i>Cocaine HCl</i> <ul style="list-style-type: none"> <li>Blue precipitate with reagent A, precipitate remains upon addition of reagent B.</li> </ul>
<b>Results</b>	<i>Cocaine Base</i> <ul style="list-style-type: none"> <li>No reaction with reagent A, blue precipitate forms upon addition of reagent B.</li> </ul>
<b>Results</b>	<i>Procaine HCl</i> <ul style="list-style-type: none"> <li>Blue precipitate with reagent A, precipitate disappears upon addition of reagent B.</li> </ul>
<b>Results</b>	<i>Tetracaine HCl</i> <ul style="list-style-type: none"> <li>Blue precipitate with reagent A, precipitate disappears upon addition of reagent B.</li> </ul>
<b>Results</b>	<i>Lidocaine HCl</i> <ul style="list-style-type: none"> <li>Blue precipitate with reagent A, precipitate disappears upon addition of reagent B.</li> </ul>
<b>Results</b>	<i>Phenyltetrahydroimidazothiazole HCl</i> <ul style="list-style-type: none"> <li>Blue precipitate with reagent A, precipitate remains upon addition of reagent B.</li> </ul>

<b>Creatine Test (Diacetyl Test)</b>	
<b>Reagent</b>	A. 0.1% aqueous solution of diacetyl B. Calcium oxide
<b>Procedure</b>	Place a few drops of the solution A into a test tube. Add sample and a pinch of calcium oxide. Immerse the test tube in a boiling water bath.
<b>Results</b>	<i>Creatine</i> Red color in solution
<b>Results</b>	<i>Creatinine</i> No reaction

<b>Dille-Kopanyi Test</b>	
<b>Reagent</b>	A. Dissolve 0.1 g cobalt acetate in 100 mL methanol and 0.2 mL glacial acetic acid. B. Dissolve 5 mL isopropylamine in 95 mL methanol.
<b>Procedure</b>	Add 2 mL solution A to powdered sample. Shake. Add 1 mL solution B. Shake.
<b>Results</b>	<i>Amobarbital</i> <ul style="list-style-type: none"> <li>A violet color indicates barbituric acid or a derivative of barbituric acid. Solution may be cloudy due to other ingredients of pharmaceutical preparations.</li> </ul>
<b>Results</b>	<i>Secobarbital</i> <ul style="list-style-type: none"> <li>A violet color indicates barbituric acid or a derivative of barbituric acid. Solution may be cloudy due to other ingredients of pharmaceutical preparations.</li> </ul>
<b>Results</b>	<i>Phenobarbital</i> <ul style="list-style-type: none"> <li>Purple</li> </ul>

<b>p-Dimethylaminobenzaldehyde (PDMAB) (Van Urk; Ehrlich)</b>	
<b>Reagent</b>	Mix 0.5% PDMAB in alcohol:concentrated hydrochloric acid (1:1). NOTE: Sulfuric acid or acetic acid may be substituted for the hydrochloric acid; the concentration of PDMAB may be modified.
<b>Results</b>	<i>LSD</i> Violet
<b>Results</b>	<i>Phencyclidine</i> Red
<b>Results</b>	<i>Dimethyltryptamine</i> Blue-violet
<b>Results</b>	<i>5-Methoxy-<math>\alpha</math>-methyltryptamine</i> Purple → Blue (2 minutes)
<b>Results</b>	<i>5-Methoxy-N,N-diisopropyltryptamine</i> Purple → Blue (2 minutes)
<b>Results</b>	<i>Bufotenine</i> Violet → Dark purple

<b>Duquenois-Levine Test</b>	
<b>Reagent</b>	A. Dissolve 2 g vanillin and 1.2 – 2.5 mL acetaldehyde in 100 mL of 95% ethanol. Store in a cool, dark place. B. Concentrated hydrochloric acid C. Chloroform
<b>Procedure</b>	Place the sample (rapid Duquenois), or an evaporated organic solvent extract of the sample, into a test tube. Add Reagent A and shake. Add Reagent B and agitate gently. Let stand for a few minutes; observe the color produced. Add Reagent C, mix gently, and let the layers separate. Note the color extracted into the chloroform.
<b>Results</b>	<i>Cannabinoids</i> <ul style="list-style-type: none"> <li>Purple/violet/blue initial color; purple/violet/blue extracted into chloroform</li> </ul>

Ferric Chloride Test		
<b>Reagent</b>	Dissolve 5 g of anhydrous ferric chloride, or 8.25 g of ferric chloride hexahydrate, in 100 mL of distilled water.  OR  Dissolve 10 g anhydrous ferric chloride, or 16.5 g of ferric chloride hexahydrate, in 100 mL of distilled water.	
<b>Procedure</b>	Dissolve the sample in ethanol, then add to the ferric chloride solution.	
<b>Results</b>	<i>Salicylates</i>	Violet
<b>Results</b>	<i>Aminopyrine</i>	Blue violet
<b>Results</b>	<i>Dipyrone</i>	Violet
<b>Results</b>	<i>Morphine</i>	Blue
<b>Results</b>	<i>Acetaminophen</i>	Blue-gray
<b>Results</b>	<i>Sodium Bicarbonate</i>	Orange
<b>Results</b>	<i>Nicotinamide</i>	Red-orange
<b>Results</b>	<i>Isonicotinamide</i>	Red-orange

Fischer-Morris Test	
Reagent	<p>A. Dissolve 1 g sodium borohydride (NaBH<sub>4</sub>) in 50 mL diglyme (commercially available).</p> <p>B. Dissolve 0.1 g p-dimethylaminocinnamaldehyde (PDMAC) in 100 mL of 0.5 N methanolic hydrochloric acid.</p>
Procedure	Mix 2 drops of solution A and 10 drops of solution B.
Results	<div style="display: flex; justify-content: space-between;"> <span><i>Methaqualone</i></span> <span>Pink</span> </div>

<b>Furfuraldehyde Test</b>	
<b>Reagent</b>	10% solution of furfuraldehyde in ethanol
<b>Procedure</b>	Dissolve the sample in ethanol, place 1 drop of the solution on a filter paper, add 1 drop of the reagent, and expose the paper to hydrochloric acid fumes for 2 to 3 minutes.
<b>Results</b>	<i>Carisoprodol</i> Violet → blue-black

<b>Froehde's Test</b>	
<b>Reagent</b>	Dissolve 50 mg of either molybdic acid, ammonium molybdate, or sodium molybdate in 10 mL of hot concentrated sulfuric acid. The freshly prepared reagent should be colorless.
<b>Results</b>	<i>Dextromethorphan</i> Blue-green
<b>Results</b>	<i>Heroin</i> Purple → Green
<b>Results</b>	<i>Morphine</i> Purple
<b>Results</b>	<i>Codeine</i> Olive → Green
<b>Results</b>	<i>Diphenhydramine</i> Yellow
<b>Results</b>	<i>Oxycodone</i> Brown-yellow
<b>Results</b>	<i>Peyote/Mescaline</i> Brown → colorless
<b>Results</b>	<i>Psilocin</i> Greenish-blue → Yellow
<b>Results</b>	<i>Psilocybin</i> Gray-blue → Green → Yellow
<b>Results</b>	<i>MDA/MDMA</i> Green → Green-black

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<b>GHB Test 1</b>	
<b>Reagent</b>	<p>A. Chlorophenol red solution: Dissolve 0.04 g chlorophenol red into 100 mL water, and adjust the solution pH to 7 with 0.1 N sodium hydroxide.</p> <p>B. Modified Schweppes solution: Dissolve 2 g dextrose into 20 mL water. Dissolve 2.4 g aniline hydrochloride into 20 mL ethanol. Mix solutions and dilute to 80 mL volume with methanol.</p> <p>C. Mix chlorophenol red solution and modified Schweppes solution in a 3:1 volume ratio (3 parts chlorophenol red to 1 part modified Schweppes).</p>
<b>Procedure</b>	<p>Add 0.5 mL of a liquid sample or a small amount of powder to a small test tube. Check the pH of liquid samples and adjust pH to a value between 5 and 7 with 0.1 N sodium hydroxide solution. Add 2 drops of the of the test reagent, and gently swirl the sample. Observe color change.</p> <p>Precaution: The reagent should be tested on tap water. If the color remains yellow, the test reagent is still effective. If tap water darkens to orange, remake the test reagent. The test reagent should be stored in a refrigerator.</p>
<b>Results</b>	<div style="display: flex; justify-content: space-between;"> <span><i>GHB/GHB</i></span> <span>Orange-red</span> </div>

<b>GHB Test 2</b>	
<b>Reagent</b>	<p>A. Bromcresol purple solution: Dissolve 0.04 g bromcresol purple into 100 mL water, and adjust the solution pH to 7 with 0.1 N sodium hydroxide.</p> <p>B. Bromthymol blue solution: Dissolve 0.04 g bromthymol blue into 100 mL water, and adjust the solution pH to 7 with 0.1 N sodium hydroxide.</p> <p>C. Modified Schweppes solution: Dissolve 2 g dextrose into 20 mL water. Dissolve 2.4 g aniline hydrochloride into 20 mL ethanol. Mix solutions and dilute to 80 mL volume with methanol.</p> <p>D. Mix bromcresol purple solution and bromthymol blue solution in a 1:1 volume ratio; the combined solution is mixed with modified Schweppes solution in a 7:1 volume ratio (7 parts of Bromcresol purple to 7 parts bromthymol blue to 1 part modified Schweppes).</p>
<b>Procedure</b>	<p>Add 0.5 mL of a liquid sample or a small amount of powder to a small test tube. Check the pH of liquid samples and adjust pH to a value between 5 and 7 with 0.1 N sodium hydroxide solution. Add 2 drops of the of the test reagent, and gently swirl the sample. Observe color change.</p> <p>Precaution: The reagent should be tested on tap water. If the color remains orange-pink, the test reagent is still effective. If tap water darkens to purple, remake the test reagent. The test reagent should be stored in a refrigerator.</p>
<b>Results</b>	<p><i>GHB/GHB</i>                      Purple</p>

<b>GHB Test 3</b>	
<b>Reagent</b>	<p>A. Bromcresol green solution: Dissolve 0.03 g bromcresol green into 100 mL 4:1 methanol-water solution (4 parts methanol to 1 part water), and adjust the solution pH to 7.0 with 0.1 N sodium hydroxide (use pH paper or test strips to measure the solution pH).</p> <p>B. Methyl orange solution: Dissolve 0.01 g methyl orange into 100 mL methanol, and adjust the solution pH to 7 with 0.1 N sodium hydroxide.</p> <p>C. Modified Schweppes solution: Dissolve 2 g dextrose into 20 mL water. Dissolve 2.4 g aniline hydrochloride into 20 mL ethanol. Mix solutions and dilute to 80 mL volume with methanol.</p> <p>D. Mix bromcresol green solution and methyl orange solution in a 1:1 volume ratio; the combined solution is mixed with modified Schweppes solution in a 3:1 ratio (3 parts Bromcresol green to 3 parts methyl orange to 1 part modified Schweppes).</p>
<b>Procedure</b>	<p>Add 0.5 mL of a liquid sample or a small amount of powder to a small test tube. Check the pH of liquid samples and adjust pH to a value between 5 and 7 with 0.1 N sodium hydroxide solution. Add 2 drops of the test reagent, and gently swirl the sample. Observe color change.</p> <p>Precaution: The reagent should be tested on tap water. If the color remains orange-pink, the test reagent is still effective. If tap water darkens to green, remake the test reagent. The test reagent should be stored in a refrigerator.</p>
<b>Results</b>	<div style="display: flex; justify-content: space-between;"> <span><i>GHB/GHB</i></span> <span>Green</span> </div>

Household Bleach		
Reagent	Commercially available	Dilute sodium hypochlorite solution
Results	<i>Cocaine Hydrochloride</i>	White streamers
Results	<i>Cocaine Base</i>	Floats
Results	<i>Procaine</i>	Dark Red



Janovsky Reagent		
<b>Reagent</b>	A. 2% m-dinitrobenzene in absolute ethanol B. 5 N potassium hydroxide	
<b>Procedure</b>	Add a few drops of reagent A to spot plate; add an equivalent amount of reagent B; add sample. Observe color change.	
<b>Results</b>	<i>Flunitrazepam</i>	Violet
<b>Results</b>	<i>Diazepam</i>	Violet
<b>Results</b>	<i>Ketamine Base</i>	Weak purple over time
<b>Results</b>	<i>Ketamine HCl</i>	Purple precipitate

Liebermann's Reagent		
<b>Reagent</b>	Dissolve 10 g potassium nitrite in sufficient sulfuric acid to produce 100 mL.	
<b>Results</b>	<i>Amphetamine</i>	Red-orange
<b>Results</b>	<i>Dextromethorphan</i>	Black
<b>Results</b>	<i>Oxycodone</i>	Bright scarlet
<b>Results</b>	<i>Peyote/Mescaline</i>	Black
<b>Results</b>	<i>Scopolamine</i>	Red-orange

Mandelin's Reagent		
<b>Reagent</b>	Dissolve 1 g ammonium vanadate in 100 mL of sulfuric acid.	
<b>Procedure</b>	Add several drops of liquid sample or a small amount of powder to a spot well plate. Add several drops of the of the test reagent and observe color change. Precaution: Always run a sulfuric acid blank when conducting this color test.	
<b>Results</b>	<i>1,4-Butanediol</i>	Brown
<b>Results</b>	<i>Amphetamine</i>	Green, darkens rapidly

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Mandelin's Reagent		
<b>Results</b>	<i>Testosterone</i>	Faint orange
<b>Results</b>	<i>Testosterone Decanoate</i>	Faint orange
<b>Results</b>	<i>Testosterone Isocaproate</i>	Slow orange
<b>Results</b>	<i>Testosterone Phenylpropionate</i>	Faint aqua
<b>Results</b>	<i>p-Methoxyamphetamine</i>	Green
<b>Results</b>	<i>Peyote/Mescaline</i>	Green → Violet-gray

Marquis Reagent		
<b>Reagent</b>	Add 8-10 drops of 40% formaldehyde solution to 10 mL concentrated sulfuric acid or 5 – 10 mL of 40% formaldehyde solution to 100 mL concentrated sulfuric acid.	
<b>Results</b>	<i>1,4-Butanediol</i>	Faint brown
<b>Results</b>	<i>2,5-Dimethoxy-4-ethylthiophenethylamine</i>	Pale orange
<b>Results</b>	<i>2,5-Dimethoxy-4-iodophenethylamine</i>	Blue
<b>Results</b>	<i>2,5-Dimethoxy-4-n-propylthiophenethylamine</i>	Pale red
<b>Results</b>	<i>4-Bromo-2,5-dimethoxyphenethylamine</i>	Green
<b>Results</b>	<i>Heroin/Morphine/Codeine</i>	Purple-violet
<b>Results</b>	<i>Methamphetamine</i>	Orange-brown
<b>Results</b>	<i>Amphetamine</i>	Orange → Brown
<b>Results</b>	<i>Hydrocodone</i>	Yellow → Brown → Violet
<b>Results</b>	<i>Oxycodone</i>	Yellow → Brown → Violet
<b>Results</b>	<i>Phentermine</i>	Orange
<b>Results</b>	<i>MDA/MDMA</i>	Purple → Black
<b>Results</b>	<i>Bufotenine</i>	Green-brown
<b>Results</b>	<i>Fentanyl</i>	Orange
<b>Results</b>	<i>Peyote/Mescaline</i>	Orange
<b>Results</b>	<i>Psilocin</i>	Greenish-brown
<b>Results</b>	<i>Psilocybin</i>	Dull orange
<b>Results</b>	<i>Diphenhydramine</i>	Yellow
<b>Results</b>	<i>Aspirin</i>	Slow pink → Rose

<b>Mecke's Reagent</b>		
<b>Reagent</b>	Dissolve 0.25 g of selenious acid in 25 mL of concentrated sulfuric acid.	
<b>Results</b>	<i>2,5-Dimethoxy-4-ethylthiophenethylamine</i>	Orange/Red/Purple
<b>Results</b>	<i>2,5-Dimethoxy-4-iodophenethylamine</i>	Brown-black
<b>Results</b>	<i>2,5-Dimethoxy-4-n-propylthiophenethylamine</i>	Orange/Red/Purple
<b>Results</b>	<i>4-Bromo-2,5-dimethoxyphenethylamine</i>	Green → Yellow (slow) → Blue (slow)
<b>Results</b>	<i>Heroin/Morphine/Codeine</i>	Dark green
<b>Results</b>	<i>Hydrocodone</i>	Yellow → Green
<b>Results</b>	<i>Peyote/Mescaline</i>	Greenish-brown → Brown
<b>Results</b>	<i>p-Methoxyamphetamine</i>	Light olive green
<b>Results</b>	<i>p-Methoxymethamphetamine</i>	Light green
<b>Results</b>	<i>MDA/MDMA</i>	Yellow → Green → Purple-black
<b>Results</b>	<i>Diphenhydramine</i>	Yellow

<b>Mercurous Nitrate</b>		
<b>Reagent</b>	Add solid sodium bicarbonate to a saturated solution of mercurous nitrate until the precipitate formed becomes yellow, then changes to a beige-like color. This reagent must be freshly prepared and shaken before use.	
<b>Procedure</b>	Dissolve the sample in alcohol, add 1 drop of the reagent, shake, and examine. May take as long as two minutes to react. An alcohol blank should be treated alongside the sample.	
<b>Results</b>	<i>Amobarbital</i>	Black
<b>Results</b>	<i>Secobarbital</i>	Black

Nitric Acid		
<b>Reagent</b>	Concentrated nitric acid	
<b>Results</b>	<i>Acetaminophen</i>	Fuming orange
<b>Results</b>	<i>Quinine</i>	Fluorescence under UV light
<b>Results</b>	<i>Quinidine</i>	Fluorescence under UV light
<b>Results</b>	<i>Heroin</i>	Lime green
<b>Results</b>	<i>Morphine</i>	Orange
<b>Results</b>	<i>Codeine</i>	Orange

Sanchez Reagent		
<b>Reagent</b>	25 mL furfural (2-furaldehyde) in 400 mL distilled water and 25 mL glacial acetic acid. Filter.	
<b>Results</b>	<i>Procaine</i>	Red
<b>Results</b>	<i>Benzocaine</i>	Red (weak)
<b>Note</b>	Test does not work well with small amounts of amines in the presence of cocaine base.	

<b>Scott's Reagent</b>	
<b>Reagent</b>	<p>A. Dissolve 2.0 g cobalt (II) thiocyanate in 50 mL distilled water then add 50 mL U.S.P. glycerin.</p> <p>B. Concentrated hydrochloric acid</p> <p>C. Chloroform</p>
<b>Procedure</b>	Place a small portion of powder in a test tube. Add solution A, note any color change. Add solution B, note any color change. Add chloroform, note color in the bottom layer (chloroform layer).
<b>Results</b>	<p><i>Cocaine Base</i></p> <ol style="list-style-type: none"> <li>1. Pink</li> <li>2. Blue → Pink</li> <li>3. Pink layer over blue layer</li> </ol>
<b>Results</b>	<p><i>Cocaine Hydrochloride</i></p> <ol style="list-style-type: none"> <li>1. Blue</li> <li>2. Pink</li> <li>3. Pink layer over blue layer</li> </ol>

<b>Modified Scott's Reagent A</b>	
<b>Reagent</b>	<p>A. Dissolve 2.0 g cobalt (II) thiocyanate in 45 mL distilled water, 5 mL glacial acetic acid and 50 mL glycerin.</p> <p>B. Concentrated hydrochloric acid</p> <p>C. Chloroform</p>
<b>Procedure</b>	Place a small portion of powder in a test tube. Add solution A, note any color change. Add solution B, note any color change. Add chloroform, note color in the bottom layer (chloroform layer).
<b>Results</b>	<p><i>Cocaine</i></p> <ol style="list-style-type: none"> <li>1. Blue precipitate</li> <li>2. Precipitate dissolves; liquid remains pink</li> <li>3. Pink layer over blue layer</li> </ol>

Sodium Nitroprusside Test	
<b>Reagent</b>	C. Dissolve 1.1 g sodium nitroprusside (sodium nitroferricyanide) in 100 mL water and 4.0 mL of acetaldehyde. Refrigerate. D. Dissolve 2.0 g sodium carbonate in 100 mL water.
<b>Procedure</b>	Place a small portion solution A in a test tube or well plate. Add a small portion of sample. Add solution B and note any color change.
<b>Results</b>	<i>Secondary Amines</i> Blue
<b>Results</b>	<i>1-(2-Methoxyphenyl)piperazine</i> Blue
<b>Results</b>	<i>1-(3-Methoxyphenyl)piperazine</i> Blue
<b>Results</b>	<i>1-(4-Methoxyphenyl)piperazine</i> Blue
<b>Results</b>	<i>Benzylpiperazine</i> Blue
<b>Results</b>	<i>Cathine</i> Rose
<b>Results</b>	<i>Cathinone</i> Rose
<b>Results</b>	<i>Methamphetamine</i> Blue
<b>Results</b>	<i>Methcathinone</i> Light blue precipitate or blue ring
<b>Results</b>	<i>p-Methoxymethamphetamine</i> Blue

Sulfuric Acid	
<b>Reagent</b>	Concentrated sulfuric acid
<b>Results</b>	<i>Testosterone</i> Fluoresces green under UV light

Van Urk Reagent (See PDMAB)	
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Wagner's Test	
<b>Reagent</b>	Dissolve 1.27 g I <sub>2</sub> into 10 mL of 0.4 mg/mL KI solution in deionized water, dilute to a volume of 100 mL with deionized water to make 0.05 M KI <sub>3</sub> .
<b>Procedure</b>	Place 1-2 mg sample in spot plate. Dissolve in 1 drop 1 N hydrochloric acid. Add 1 drop of KI <sub>3</sub> solution.
<b>Results</b>	<i>Diazepam</i> Brown-black precipitate, brown solution
<b>Results</b>	<i>Scopolamine</i> Brown → Black

Walt's Test	
<b>Reagent</b>	A. Dissolve 10 g ammonium acetate in 100 mL deionized water. Add 40 mL concentrated acetic acid. B. Dissolve 2.0 g cuprous chloride (CuCl) in 100 mL of 10% ammonium acetate buffer.
<b>Procedure</b>	Place 1-2 mg sample in spot well. Add four drops 1N NaOH prepared in methanol, wait 60 seconds. Add one drop of solution B.
<b>Results</b>	<i>Flunitrazepam</i> Light green

Zwicker's Test	
<b>Reagent</b>	A. 0.5% aqueous solution of copper sulfate B. Chloroform containing 5% by volume of pyridine
<b>Procedure</b>	Add approximately 0.5 mL of solution A to a test tube. Add a small amount of sample. Mix gently and add an equal volume of solution B. Shake, let the layers separate.
<b>Results</b>	<i>Amobarbital</i> Violet in chloroform layer
<b>Results</b>	<i>Secobarbital</i> Violet in chloroform layer
<b>Results</b>	<i>Phenobarbital</i> Purple in chloroform layer, very weak blue with the addition of 1 drop of glacial acetic acid



## Appendix 1E – Crystal and Precipitate Test Reagent Preparation and Procedures

Results listed below are not comprehensive. Additional results and reagents can be found in the references list in ADM 1-11.

Acetic Acid	
<b>Reagent</b>	1:50 solution of acetic acid in 3% ammonia
<b>Results</b>	<i>Amobarbital</i> Long, branching needles and hexagonal plates

Ammoniacal Nickel Acetate	
<b>Reagent</b>	Mix one volume of 5% nickel acetate solution with one volume of 10% NH <sub>4</sub> OH.
<b>Procedure</b>	Add one drop of the reagent to about 1 mg of sample.
<b>Results</b>	<i>Phenobarbital</i> Single rectangular crystals

Barium Chloride or Barium Nitrate Precipitate Test	
<b>Reagent</b>	12 g of barium chloride in 100 mL of distilled water OR 6.5 g of barium nitrate in 100 mL of distilled water
<b>Procedure</b>	Add reagent to a test tube. Add a small portion of sample.
<b>Results</b>	<i>Sulfates</i> White precipitate

<b>Bismuth Iodide in Diluted Sulfuric Acid Solution</b>	
<b>Reagent</b>	Dissolve 50 g bismuth subnitrate ( $\text{Bi}(\text{NO}_3)_3$ ) in 70 mL diluted $\text{HNO}_3$ (1:1 with water) and dilute to 100 mL with water (stock prepared fresh daily). Dissolve 1.25 g KI in 2 mL water, and add 2.5 mL $\text{H}_2\text{SO}_4$ and 0.5 mL stock concentrated $\text{Bi}(\text{NO}_3)_3$ solution.
<b>Procedure</b>	Utilize the hanging drop technique.
<b>Results</b>	<p><i>Methamphetamine</i></p> <ul style="list-style-type: none"> <li>Drops, long orange splinters, blades, needles; also deep red angular grains (red prisms only after evaporation): drops crystallizing in orange-red prisms with conspicuously slanting ends; also "mossy" formation of grains and some large deep red grains.</li> </ul>
<b>Results</b>	<p><i>Phentermine</i></p> <ul style="list-style-type: none"> <li>Red rods with drying dendritic clusters</li> </ul>

<b>Copper Sulfate Precipitate Test</b>	
<b>Reagent</b>	A. 1% copper sulfate solution B. Concentrated sulfuric acid
<b>Procedure</b>	Place 3 drops of reagent A in a spot well; add 7 drops of reagent B. Add a small portion of test sample.
<b>Results</b>	<i>Bromide</i> Purple/Black precipitate

<b>Fulton's Iodine Reagent C-2</b>	
<b>Reagent</b>	Mix 3 mL of 1 g KI and 1 g $\text{I}_2$ in 100 mL water, 4.4 mL saturated iodine solution in 2:1 acetic acid, 0.5 mL water and 3.8 mL 1:3 $\text{H}_2\text{SO}_4$ .
<b>Results</b>	<i>Hydrocodone</i> Single rods and rosettes of needles which grown into rods

Gold Bromide	
<b>Reagent</b>	Dissolve 1 g gold chloride ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ ) in 1.1 mL HBr and 1 mL $\text{H}_2\text{O}$ and 17.5 mL $\text{H}_2\text{SO}_4$ .
<b>Procedure</b>	Dilute 2 parts solution with 3 parts $\text{H}_2\text{O}$ to produce a final volume of 30 mL.
<b>Results</b>	<i>Amphetamine</i> Trapezoidal blades or small red cigars

Gold Chloride	
<b>Reagent</b>	Dissolve 1 g gold chloride ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ ) in 20 mL $\text{H}_2\text{O}$ .
<b>Procedure</b>	Place a small amount of powder on a slide, dissolve with diluted $\text{H}_2\text{SO}_4$ (1:1 with water). Add a drop of reagent. Use a small probe to immerse in reagent and draw the probe through the dissolved sample.
<b>Results</b>	<i>Cocaine</i> <ul style="list-style-type: none"> <li>Serrated needles, long thin combs or ladders with branches, very characteristic</li> </ul>
<b>Results</b>	<i>Amphetamine</i> <ul style="list-style-type: none"> <li>Thin, flat, feathery, leaf shaped crystals, low birefringence; some X's and thin birefringent rods</li> </ul>

Gold Chloride in Diluted Phosphoric Acid Solution	
<b>Reagent</b>	Dissolve 1 g gold chloride ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ ) in 20 mL diluted $\text{H}_3\text{PO}_4$ (1:2 with water).
<b>Procedure</b>	Utilize the direct addition or hanging drop technique.
<b>Results</b>	<i>Methamphetamine</i> <ul style="list-style-type: none"> <li>Long blades and jointed crystals, fairly high birefringence</li> </ul>
<b>Results</b>	<i>Phentermine</i> <ul style="list-style-type: none"> <li>Long serrated blades often spearheaded and some plates</li> </ul>

<b>Iodine-Potassium Iodide Reagent M-2</b>	
<b>Reagent</b>	Mix 1.0 mL of iodine-potassium iodide (5 g iodine and 30 g potassium iodide in 100 mL of water), 1.5 mL concentrated hydrochloric acid and 1.5 mL phosphoric acid.
<b>Results</b>	<i>Oxycodone</i> <ul style="list-style-type: none"> <li>Brown-red varnish fixed to deposit rim and dark red to black grains</li> </ul>

<b>Iodine-Potassium Iodide Reagent N-1</b>	
<b>Reagent</b>	Mix 0.5 mL of iodine-potassium iodide (10 g iodine and 35 g potassium iodide in 100 mL of water), 1.8 mL glacial acetic acid, 1.5 mL water, and 2.2 mL phosphoric acid.
<b>Results</b>	<i>Oxycodone</i> <ul style="list-style-type: none"> <li>Brown-red varnish fixed to deposit rim and dark red to black grains and brown rods that are birefringent, with negative elongation and slight negative absorption</li> </ul>

<b>Mercuric Chloride</b>	
<b>Reagent</b>	Dissolve 0.5 g mercuric chloride in 10 mL of water.
<b>Results</b>	<i>Heroin</i> Fine dendrites

<b>Mercuric Iodide</b>	
<b>Reagent</b>	Make a saturated solution of mercuric iodine in 10% hydrochloric acid (leave some undissolved crystals of HgI <sub>2</sub> ).
<b>Results</b>	<i>Heroin</i> Clusters of needles and threads; also blades in clusters and dendrites

Platinic Bromide in Acetic Acid and Sulfuric Acid	
<b>Reagent</b>	Add 1 part glacial acetic acid to 3 parts platinic bromide in dilute (2:3 with water) sulfuric acid.
<b>Procedure</b>	Add reagent to sample without cover glass.
<b>Results</b>	<i>Oxycodone</i> Blade crystals growing in clusters from the rim of the deposit

Platinic Bromide in Hydrobromic Acid and Sulfuric Acid	
<b>Reagent</b>	Mix equivalent amounts of platinic bromide in 40% HBr and platinic bromide in dilute (2:3 with water) sulfuric acid.
<b>Procedure</b>	Add reagent to an aqueous drop and evaporate.
<b>Results</b>	<i>Oxycodone</i> Large dendritic clusters of narrow orange bladed crystals
<b>Results</b>	<i>Hydrocodone</i> Rosettes of yellow needles

Platinic Bromide in Sulfuric Acid	
<b>Reagent</b>	Dissolve 1 g platinic chloride ( $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ ) in 1.7 mL 40% HBr. Dilute to 20 mL with 2 parts concentrated sulfuric acid and 3 parts water.
<b>Procedure</b>	Add reagent to an aqueous drop and evaporate.
<b>Results</b>	<i>Oxycodone</i> Clusters of needles and narrow orange blades with slight dichroism

Platinic Chloride	
<b>Reagent</b>	Dissolve 5 g platinic chloride ( $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ ) in 100 mL water.
<b>Procedure</b>	Place small amount of powder on slide and add a drop of reagent.
<b>Results</b>	<i>Cocaine</i> Delicate, feathery crystals
<b>Results</b>	<i>Amphetamine</i> Needles, narrow irregular blades of low birefringence
<b>Results</b>	<i>Heroin</i> Small dark needles formed into a burr

Platinic Chloride in Diluted Phosphoric Acid Solution	
<b>Reagent</b>	Dissolve 1 g platinic chloride ( $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ ) in 20 mL diluted $\text{H}_3\text{PO}_4$ (1:2 with water).
<b>Procedure</b>	Use the hanging drop technique.
<b>Results</b>	<i>Methamphetamine</i> <ul style="list-style-type: none"> <li>Grains with sharp edges which aggregate in chains and short prisms; birefringent</li> </ul>
<b>Results</b>	<i>Phentermine</i> <ul style="list-style-type: none"> <li>Yellow plates, square cut or elongated hexagons with low birefringence</li> </ul>

Platinic Iodide	
<b>Reagent</b>	Dissolve 1 g platinic iodine and 25 g sodium iodide in 100 mL water.
<b>Procedure</b>	Dissolve sample in 30% acetic acid.
<b>Results</b>	<i>Ketamine</i> Rhomboidal plates with rosettes of plates over time

Potassium Permanganate	
<b>Reagent</b>	Dissolve 0.5 g potassium permanganate ( $\text{KMnO}_4$ ) in 20 mL water; add one drop of concentrated $\text{H}_3\text{PO}_4$ .
<b>Procedure</b>	Dissolve unknown with several drops of 30% glacial acetic acid in water. Add one drop of reagent and let stand.
<b>Results</b>	<i>Phencyclidine</i> Bow-tie shaped crystals
<b>Results</b>	<i>Methaqualone</i> Plates

Silver Nitrate Crystal Test	
<b>Reagent</b>	Dissolve 0.1 g silver nitrate into 10 mL water.
<b>Procedure</b>	Add 1 drop of reagent to 1 drop of sample.
<b>Results</b>	<i>GHB</i> Rectangular crystals

Silver Nitrate Precipitate Test	
<b>Reagent</b>	A. 5% silver nitrate solution B. Concentrated nitric acid C. Concentrated ammonium hydroxide
<b>Procedure</b>	1. Dissolve 1 -5 mg of sample in 1 -2 mL of distilled water in a test tube. Prepare two of these solutions. 2. Add several drops of reagent A to both test tubes. Observe any precipitate formed. 3. (Optional) Remove excess water from both test tubes. Add several drops of reagent B to the first test tube and several drops of reagent C to the second test tube.
<b>Results</b>	<i>Chloride</i> <ul style="list-style-type: none"> <li>White precipitate that is insoluble in reagent B, but soluble in reagent C</li> </ul>
<b>Results</b>	<i>Bromide</i> <ul style="list-style-type: none"> <li>Cream precipitate that is insoluble in reagent B, but soluble in reagent C</li> </ul>
<b>Results</b>	<i>Iodide</i> <ul style="list-style-type: none"> <li>Yellow precipitate that is insoluble in reagent B and turns white but remains insoluble in reagent C</li> </ul>
<b>Results</b>	<i>Phosphate</i> <ul style="list-style-type: none"> <li>Yellow precipitate that is soluble in reagent B</li> </ul>
<b>Results</b>	<i>Citrate</i> <ul style="list-style-type: none"> <li>White precipitate that is soluble in reagent B</li> </ul>
<b>Results</b>	<i>Tartrate</i> <ul style="list-style-type: none"> <li>White precipitate that is soluble in reagent B</li> </ul>

Sodium Carbonate	
<b>Reagent</b>	Dissolve 5 g sodium carbonate (Na <sub>2</sub> CO <sub>3</sub> ) in 100 mL of H <sub>2</sub> O.
<b>Results</b>	<i>Methaqualone</i> Needles

Sulfuric Acid	
<b>Reagent</b>	1 M sulfuric acid solution
<b>Procedure</b>	Place one drop of sulfuric acid solution on a glass microscope slide. Add a few crystals of sample to the drop. After any effervescence dissipates, place the slide under microscope.
<b>Results</b>	<i>Calcium</i> <ul style="list-style-type: none"> <li>Thin needle-like crystals observable at 100X power under a light microscope</li> </ul>

Uranyl Acetate	
<b>Reagent</b>	8.5% uranyl acetate in 6% acetic acid solution
<b>Procedure</b>	Place one drop of uranyl acetate solution on a glass microscope slide. Add a few crystals of sample to the drop. After any effervescence dissipates, place the slide under microscope.
<b>Results</b>	<i>Sodium</i> <ul style="list-style-type: none"> <li>Tetrahedral crystals observable at 100X power under a light microscope</li> </ul>
<b>Results</b>	<i>Potassium</i> <ul style="list-style-type: none"> <li>Rod-like crystals observable at 100X power under a light microscope</li> </ul>



**Revision:** 4

**Issue Date:** September 5, 2019

**Effective Date:** September 9, 2019

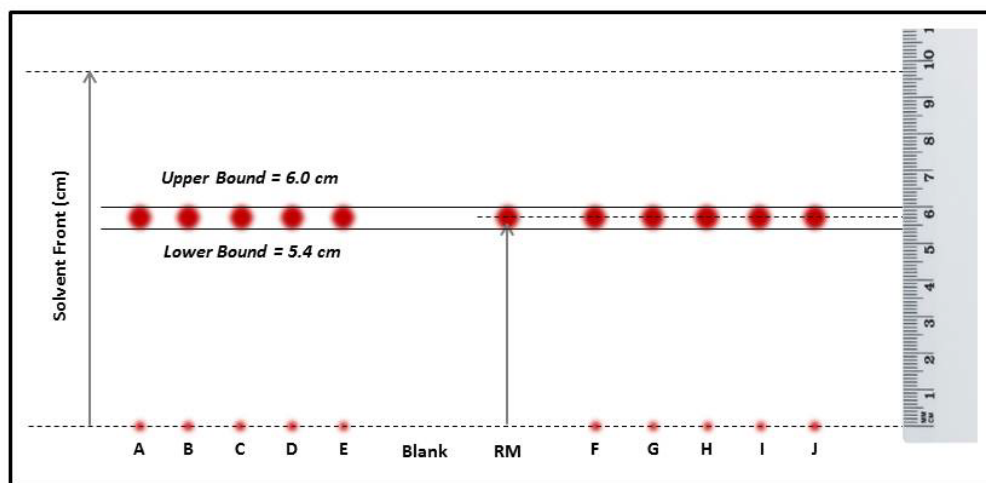
**Approved By:** Nelson A. Santos

Wagenaar's Reagent	
<b>Reagent</b>	Add ethylenediamine to a 5% solution of copper sulfate until the solution becomes dark violet.
<b>Procedure</b>	Place a drop of reagent in contact with a small amount of the dry sample.
<b>Results</b>	<i>Phenobarbital</i> Large number of small rectangular prisms
<b>Results</b>	<i>Amobarbital</i> Light blue needles in clusters
<b>Results</b>	<i>Secobarbital</i> Large rosettes of fine needles

**Effective Date:** September 9, 2019

**Approved By:** Nelson A. Santos

### Calculating Retention (Retardation) Factors



Calculate the retention (retardation) factor ( $R_f$ ) for the reference material (RM) as follows:

$$R_f(\text{RM}) = \frac{\text{Distance traveled by RM}}{\text{Distance traveled by solvent}} = \frac{5.7 \text{ cm}}{9.7 \text{ cm}} = \mathbf{0.59}$$

**Lower bound** =  $R_f$  (RM)  $\times$  Distance traveled by solvent  $\times$  95%

$$= 0.59 \times 9.7 \text{ cm} \times 0.95 = \mathbf{5.4 \text{ cm}}$$

**Upper bound** =  $R_f$  (RM)  $\times$  Distance traveled by solvent  $\times$  105%

$$= 0.59 \times 9.7 \text{ cm} \times 1.05 = \mathbf{6.0 \text{ cm}}$$

## TLC Visualization Methods and Reagents

For TLC system details (solvent systems, stationary phases) see SWGDRUG. The target analytes and systems listed below are not comprehensive. Additional TLC information can be found in the references listed in ADM 1-11.

Target Analyte	TLC Systems
Amobarbital	7, 12
Amphetamine	8
Cocaine	13, 14
Codeine	3
Diazepam	7, 11
Dimethyltryptamine	18
Flunitrazepam	7, 11
Heroin	3, 4, 5, 13
Hydrocodone	13, 14, 15
Ketamine	5, 6
LSD	1, 2,
Marijuana	9, 10, 13, 20
MDMA	5, 6
Methamphetamine	5, 6
Methaqualone	5, 6, 7
Morphine	3, 4
Noscapine	4
Opium	3, 4
Oxycodone	5, 6, 18, 19
Papaverine	3, 4
Phencyclidine	11, 16, 17
Phenobarbital	4, 7
Phentermine	5, 6
Secobarbital	7, 12
Testosterone (and esters)	19

**Reagents:**

<b>Acidified Potassium Permanganate</b>	
<b>Reagent</b>	1% potassium permanganate in 0.25 M sulfuric acid

<b>2,6-Dichloroquinone-4-chloroimide</b>	
<b>Reagent</b>	1% solution of 2,6-dichloroquinone-4-chloroimide in methanol

<b>Dragendorff</b>	
<b>Reagent</b>	A. Mix together 2 g bismuth subnitrate, 25 mL glacial acetic acid, 100 mL water B. Dissolve 40 g potassium iodide in 10 mL water Mix 10 mL solution A, 10 mL solution B, 20 mL glacial acetic acid, and 100 mL water (prepare every 2 days)

<b>Fast blue 2B salt</b>	
<b>Reagent</b>	15 mg fast blue 2B salt in 20 mL methanol

<b>Iodoplatinate/ Acidified Iodoplatinate</b>	
<b>Reagent</b>	Dissolve 0.25 g platonic chloride and 5 g potassium iodide in 100mL water. To make acidified iodoplatinate solution: Add 5 mL hydrochloric acid to 100 mL iodoplatinate solution.

<b>Marquis</b>	
<b>Reagent</b>	Add 8-10 drops of 40% formaldehyde solution to 10 mL concentrated sulfuric acid.

<b>Mercurous Nitrate</b>	
<b>Reagent</b>	Saturated solution of mercurous nitrate

1% Ninhydrin	
Reagent	1% ninhydrin in methanol

0.5% p-dimethylaminobenzaldehyde (PDMAB)	
Reagent	0.5% PDMAB in alcohol: concentrated hydrochloric acid (1:1)

Van Urk's	
Reagent	Dissolve 1 g p-dimethylaminobenzaldehyde in 100 mL ethanol, add 10 mL hydrochloric acid

## Appendix 1G – Qualitative Method Modifications

All modifications must be documented in the casefile.

- A. Modifications to qualitative methods that are not expected to negatively impact the data are permitted without further action, provided they enhance the method.

For example:

- Lower the split ratio.
- Change in the sample solvent (except NMR).
- Increase the injection volume.
- Shorten the solvent delay.
- Extend the hold time at the end of a method.
- Increase the number of scans (IR, Raman, and NMR only).
- Change the threshold or gain to improve sensitivity.

NOTE: Any modifications made to decrease the sensitivity or to limit the detection of certain analytes require supervisory approval (e.g., extended solvent delay to not detect a specific analyte).

- B. The following modifications to qualitative methods require a check of the method:

- Trimming of a column.
- Replacement of a column with another one having the same properties and dimensions.

NOTE: Solvent delay may be adjusted as needed, provided the earliest eluting compound is detected.

- The method check includes:
  - A single injection of the repeatability mixture from validation (1-3.1.2.1)
  - Evaluation of the data per selectivity acceptance criteria (1-3.1.1.2)

NOTE 1: Full method revalidation or column change may be required if the early eluting compound retention factors or retention time falls below the acceptable range.

NOTE 2: Positive controls may need to be rerun if retention times of mixture are not within acceptance criteria of previous month's values.

- Documentation of the check and results in the instrument logbook

C. The following changes to qualitative methods result in a new method and require a new method name and complete validation:

- Change in gradient (i.e., temperature, buffer ratio, voltage, flow).
- Change in detector parameters (i.e., scan rate on MS, resolution on IR).
- Installation of a column with different properties, dimensions, or technology.
- Implementation of a different buffer composition (e.g., phosphate buffer changed to acetate buffer or pH 3 changed to pH 5).
- Change in the scan range.
- Change in carrier gas.

NOTE: For instrument modifications and required performance verification procedures see 1-6.

### Appendix 1H – Analytical Supplies and Services

Providers (vendors) of analytical supplies and services must be evaluated to ensure they meet the requirements listed below.

<b>Consumables</b>		
<b>Item(s)</b>	<b>Vendor Requirement(s)</b>	<b>Additional Notes</b>
Reference Materials	Accreditation through: ISO/IEC 17025 ISO 17034	
<b>Services</b>		
Balance Calibration	Accreditation through ISO/IEC 17025	Required annually; ensure scope includes the type of balance(s) to be calibrated
Weight Calibration	Accreditation through ISO/IEC 17025	Required annually
<b>Supplies</b>		
External Proficiency Samples	Accreditation through ISO/IEC 17043	
Reagents		See 1-11



## **Appendix 2A – Random Sampling Procedures**

- Random sampling procedures are required when selecting units from an exhibit (i.e., the population) for net weight determination, qualitative analysis, and composite formation.
- Random selection processes are used to ensure:
  - All units in a population have an equal chance of being selected.
  - Selection bias is avoided.
- Random selection:
  - Allows the use of statistical methods to analyze sample results.
  - Allows inferences to be made on the population.

### **Method 1: Random number generator (RNG)**

- Arrange or stack all population units in a pattern.
- Open the RNG and enter the total number of units in the exhibit and the number of units to be selected.
- Identify the units to be sampled by following a row or a column starting at the upper left corner.,
- Segregate or label (if possible) the units selected (unit 1, unit 2, unit 3, etc.)

### **Method 2: Lottery Method A**

- Place units into one or more containers (bowls, bags, etc.).
- Mix the units thoroughly.

OR

- Randomly arrange or stack all population units in a pattern (columns, rows, or both).
- Perform 'blind' selection by removing one unit at a time.
- Segregate or label (if possible) the units selected (unit 1, unit 2, unit 3, etc.).

### **Method 3: Lottery Method B**

- Arrange or stack all population units in a pattern (columns, rows, or both).
- Place numbered pieces of paper (or balls, marbles, etc.) in a container (bowl, bag, etc.).

- Mix the numbers thoroughly.
- Perform 'blind' selection by reaching into the container and removing one piece of paper (or ball, marble, etc.) at a time.
- Identifies the units to be sampled by following a row or a column starting at the upper left corner of the stacked or arranged units.
- Segregate or label (if possible) the units selected (unit 1, unit 2, unit 3, etc.).

## Appendix 2B – Reporting Statements

### Department of Justice (DOJ) Uniform Language for Testimony and Reports (ULTR) reference statement

The terminology used in the preparation of this report is consistent with the current Department of Justice Uniform Language For Testimony and Reports For General Forensic Chemistry and Seized Drug Examinations.

#### Net Weight Determination Statements for Remarks Section of DEA-113 form:

##### Direct weighing

The net weight was determined by direct weighing of all unit(s). The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

##### Extrapolation

The net weight is an extrapolated value based on the individual weights of [#] units. The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

**OR**

The net weight is an extrapolated value based on the weights of [#] groups of [#] units each. The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

##### Combination (Direct and Extrapolation)

The net weight is the combination of the direct weight of [#] units and an extrapolated value based on the individual weights of [#] units. The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

##### Subgroups

The net weight is an extrapolated value based on the individual weights of subgroups of [#], [#], and [#] units. The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

##### Exemplars

The net weight was determined by direct weighing of all unit(s). No net weight uncertainty reported.

**OR**

The net weight was determined by direct weighing of all unit(s). The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.

**OR**

The net weight is the combination of the direct weight of [#] units and an extrapolated value based on the individual weights of [#] units. No net weight uncertainty reported.

### Net Weight Determination Statements for Remarks Section of DEA-113 form:

#### Liquids and Dosage Units (additional remark)

Total volume = X mL (net); X mL (reserve); substance concentration: Y mg/mL.

**OR**

Total dosage unit count = X [Units] (net); X [Units] (reserve); substance concentration: Y mg/[unit].

### Identification Statements for Remarks Section of DEA-113 form:

#### Scenario A: 1-9 units – One or more substances confirmed in all units tested

[List Substance(s) Identified] confirmed in [#] units tested of [#] units received. A composite [where applicable] was formed from [#] units for further testing. [List Substance(s) Identified] also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

**Single unit (composite formed):** A composite was formed from 1 unit for testing of 1 unit received. [List Substance(s) Identified] confirmed in the composite. Salt form [and/or] isomer determined from testing the composite.

**Single unit (no composite formed):** [List Substance(s) Identified] confirmed in 1 unit tested of 1 unit received. Salt form [and/or] isomer determined from testing 1 unit.

#### Scenario B: 10 or more units – One or more substances confirmed in all units tested

[List Substance(s) Identified] confirmed in [#] units tested of [#] units received indicating, to at least a 95% level of confidence, that at least 90% of the units in the population contain the substance(s). A composite [where applicable] was formed from [#] units for further testing. [List Substance(s) Identified] also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

#### Scenario C: 10 or more units – One or more substances confirmed in all units tested and additional controlled substance(s) confirmed in some but not all units tested.

[List Substance(s) Identified] confirmed in [#] units tested of [#] units received indicating, to at least a 95% level of confidence, that at least 90% of the units in the population contain the substance(s). [List controlled substance(s)] also confirmed in [#] of the [#] units tested. A composite [where applicable] was formed from [#] units for further testing. [List Substance(s) Identified] also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

#### Scenario D: 60 or more units – One negative result observed

[List Substance(s) Identified] confirmed in [#] of [#] units tested of [#] units received indicating, to at least a 95% level of confidence, that at least 84% of the units in the population contain the substance(s). A composite [where applicable] was formed from [#] units for further testing. [List Substance(s) Identified] also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

#### Scenario E: 10 or more units – Two or more negative results observed

[List Substance(s) Identified] confirmed in [#] of [#] units tested of [#] units received. A composite [where applicable] was formed from [#] units for further testing. [List Substance(s) Identified] also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

### Identification Statements for Remarks Section of DEA-113 form:

**Scenario F:** No reportable analytes identified or data insufficient for confirmation

No controlled substance(s) identified in [#] unit(s) tested of [#] unit(s) received. A composite *[where applicable]* was formed from [#] units for further testing. *[List Substance(s) Identified]* confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

**Single unit (composite formed):** A composite was formed from 1 unit for testing of 1 unit received. No controlled substance(s) identified in the composite.

**Scenario G:** Arbitrary Sampling:      X.01 – Analyzed unit(s)      X.02 – Unanalyzed unit(s)

X.01 *[List Substance(s) Identified]* confirmed in [#] unit(s) tested. A composite *[where applicable]* was formed from [#] unit(s) for further testing. *[List Substance(s) Identified]* also confirmed in the composite. Salt form [and/or] isomer determined from testing {[#] units/the composite}.

X.02 No analysis per *[Enter approval source]*.

NOTE 1: *[List Substance(s) Identified]* should only include the base form of the substance(s) identified and should not include any salt form or isomer designations (e.g., Cocaine not Cocaine Base).

NOTE 2: For salt form and isomer statements, edit statements to accurately reflect analysis conducted.

### Purity Statements for Remarks Section of DEA-113 form:

**Scenario H:** To be used:

- (1) with representative mixtures (i.e., composites, single unit liquids) **and**
- (2) when required minimum sample amounts are used (Table 2).

Purity determined from testing the composite; the purity and amount pure substance values are representative of the entire exhibit. All uncertainty values represent expanded uncertainty estimates at the 95% level of confidence.

**Scenario I:** To be used:

- (1) with representative mixtures (i.e., composites) **but**
- (2) when required minimum sample amounts are **not** used (Table 2).

Purity determined from testing the composite; the purity and amount pure substance values are not representative of the entire exhibit. All uncertainty values represent expanded uncertainty estimates at the 95% level of confidence.

**Scenario J:** To be used:

- (1) with non-representative mixtures (e.g., gummy),
- (2) regardless of sample amount used.

Purity determined from testing a non-representative portion of the exhibit; the purity and amount pure substance values are not representative of the entire exhibit. All uncertainty values represent expanded uncertainty estimates at the 95% level of confidence.

## **Appendix 2C – Composite Formation Procedures**

When the formation of a composite is required, one of the following options will be chosen:

Option 1: The Forensic Chemist (FC) combines all units in the exhibit.

Option 2: The FC performs incremental sampling to produce a primary sample (composite) that is representative of the entire exhibit (sampling target).

### **Option 1: Combining all units in an exhibit**

- If practical, the FC forms the composite by combining the entire contents of all units in the exhibit (including untested units, if present).
  - The original appearance of solid dosage form exhibits (except capsules) must be documented via photograph.

NOTE: The original appearance of the exhibit may be documented via photograph.

- For powders, crystalline materials, body carries, and dosage units, the resulting composite is ground, sieved to a maximum particle size of 850 µm (20-mesh), and mixed thoroughly.

NOTE: Moist materials that are mixed and ground, but unable to pass through a 20-mesh sieve are considered representative composites.

- For liquids, the resulting composite is mixed thoroughly.

### **Option 2: Performing incremental sampling**

- Powders, crystalline materials, and body carries
  - Units containing at least 1 g of material:
    - One increment is approximately 1 g.
    - Form a minimum 15-g composite.
    - Remove at least 15 randomly selected increments from as many units as possible, considering all units in the exhibit.

NOTE 1: If exhibit contains less than 15 units, some units are sampled more than once.

- Combine all increments to form the composite.
- Grind, sieve to a maximum of 850 µm (20-mesh), and mix thoroughly.

NOTE: Moist materials that are mixed and ground, but unable to pass through a 20-mesh sieve are considered representative composites.

- Units containing less than 1 g of material:
  - One increment = one unit.
  - Form a composite of sufficient size to complete the required analysis (i.e., salt and isomer testing, qualitative verification, quantitation) as well as a subsequent reanalysis, if necessary.
  - Remove at least 15 randomly selected increments, considering all units in the exhibit.  
  
NOTE 1: For exhibits containing 15 units or less, combine all units (**Option 1**).
  - Combine all increments to form the composite.
  - Grind, sieve to a maximum particle size of 850 µm (20-mesh), and mix thoroughly.  
  
NOTE: Moist materials that are mixed and ground, but unable to pass through a 20-mesh sieve are considered representative composites.
- Units containing both < 1 g and > 1 g of material:
  - One increment = entire contents of the selected units if the selected unit contains < 1 g and one (or more) 1-g increment if the selected unit contains > 1 g.
  - Form a composite of sufficient size to complete the required analysis (i.e., salt and isomer testing, qualitative verification, quantitation) as well as a subsequent reanalysis, if necessary.
  - Remove at least 15 randomly selected increments (or more, if needed), considering all units in the exhibit.  
  
NOTE: Selected units containing >1 g of material may have to be sampled more than once.
  - Combine all increments to form the composite.
  - Grind, sieve to a maximum particle size of 850 µm (20-mesh), and mix thoroughly.  
  
NOTE: Moist materials that are mixed and ground, but unable to pass through a 20-mesh sieve are considered representative composites.
- Liquids and Solutions
  - One increment = 1 mL (or entire unit if < 1 mL)
  - Remove at least 15 randomly selected increments from as many units as possible, considering all units in the exhibit.

NOTE: For exhibits containing 15 units or less, combine all units (**Option 1**) or remove one increment from each unit.

- Combine all increments to form the composite.
- Mix thoroughly.
- Solid Dosage Forms
  - One increment = One dosage unit or one partially remaining unit after testing
  - Remove at least 15 randomly selected increments, considering all units in the exhibit.

NOTE: For exhibits containing 15 units or less, combine all units (**Option 1**).

- Combine all increments to form the composite.
- Grind, sieve to a maximum particle size of 850  $\mu\text{m}$  (20-mesh), and mix thoroughly.



## Appendix 2D – Methamphetamine Isomer Reporting

The Forensic Chemist (FC):

- Determines the isomer using an appropriate separation technique.
- Calculates the relative percentage of l-methamphetamine enantiomer in the sample (composite) using the following equation:

$$\frac{\text{l-methamphetamine peak area}}{\text{d-methamphetamine peak area} + \text{l-methamphetamine peak area}} \times 100 = \% \text{ l-methamphetamine}$$

NOTE: The relative % of l-methamphetamine can be obtained directly from the integration table as “Area %” if the only peaks integrated are those of d- and l- methamphetamine.

- Reports the d-isomer of methamphetamine on the DEA-113 when:
  1. The purity of total methamphetamine hydrochloride in the exhibit is  $\geq 80\%$ .
  2. The relative percentage of l-isomer (l-methamphetamine) is  $< 1\%$ .

NOTE: In all other instances, the isomer of methamphetamine is not reported.

- If not automatically calculated by the instrument software, records any manual calculations of the % of l-methamphetamine in the CDR or attachments.

## **Appendix 2E – Procedures for the Germination and Analysis of Marijuana Seeds**

### **1 Sampling**

- Visually evaluate the seeds to determine if more than one population is present (i.e., multiple seed types). Sub-exhibit populations accordingly.
- Determine the total number of seeds present in the population either by direct count or by unit count extrapolation.
- Randomly select half of the seeds from each population (up to 29 seeds) for germination. No attempt should be made to purposefully exclude damaged seeds.
- Retain the remaining seeds.

### **2 Germination and Transplantation**

- Germinate and grow the seeds in a secure locker in the in-process vault or other secured area with controlled access.
- Use the following supplies during the germination of the seeds:
  - Seeds (half of the seeds in the exhibit up to a maximum of 29)
  - Water (room temperature)
  - Sealable plastic container or bag
  - Paper towels
  - Aluminum foil
  - Transplant/growth supplies:
    - Forceps
    - Seed starting soil
    - Small plant pots with drainage (e.g., 3" size)
    - Water
    - Fertilizer
    - Artificial light source (e.g., 400 W high pressure sodium lamp)
- Germination Procedure:

- Place the selected seeds on a damp paper towel and seal the folded paper towel in a plastic container.
- Place the container in a dark environment.
- The container should be kept in the dark for 3-5 days. Monitor the seeds to ensure they do not dry out. Once a root has emerged from the seed, the seed is considered to have germinated.
- Transfer all germinated seed(s) (using forceps) to small pots containing soil, planting one seed per pot.
- Keep the soil moist.
- Plant fertilizer may be added to the water to encourage growth.
- Use an artificial light source. Place the light at least two feet above the plants to avoid excess heating.
- Grow the plants to a sufficient height to obtain enough plant material for testing.

### **3 Identification**

- Cannabis is a single stem plant, and each stalk supported by its own root system will be considered one plant.
- At minimum, two samples are removed from each plant for testing.

NOTE: Retain remaining plant material as reserve evidence in a manner to prevent degradation.

- The identification of marijuana is completed in accordance with 2-5.8.1.

## Appendix 2F – Uncertainty of Measurement Estimates

### 1.1 Net Weight

The uncertainty associated with net weight measurements is affected by factors including but not limited to those listed below:

- The number of weighing operations used to obtain a weight
- The process by which a weight is obtained (direct or extrapolation)
- Operator differences
- Balance type
- Balance readability
- Balance calibration
- Balance accuracy
- Balance location and operating environment
- Balance long-term performance

For each balance, mass uncertainty ( $u_{mass}$ ) values are established to incorporate these factors.  $u_{mass}$  is described by Equation 1, where  $u_{bal}$  is the balance calibration uncertainty,  $u_{process}$  is the process uncertainty obtained from one year of performance verification measurements (at high-load), and  $u_{acc}$  is the uncertainty associated with the reference weights used during those high-load measurements.

$$u_{mass} = \sqrt{u_{bal}^2 + u_{process}^2 + u_{acc}^2}^{**}$$

Equation 1

The balance calibration component ( $u_{bal}$ ) captures differences in balance manufacturers, calibration procedures, reference standard weights, and calibration personnel. The  $u_{process}$  component incorporates variations resulting from balance location, environment, operator, and performance verification procedures. The  $u_{acc}$  component captures the uncertainty associated with the accuracy of the in-house reference weights used throughout the laboratory system.

The table below lists the system-wide mass uncertainty ( $u_{mass}$ ) values established for each type of balance available for net weight measurements. Values are calculated using the mean  $u_{mass}$  value per balance type plus 3 standard deviations. These values must be used when calculating the uncertainty associated with net weights obtained either by direct measurement or by extrapolation.

<u>Readability (g):</u>	<u><math>u_{mass}</math> (g):</u>
0.1	0.26028
0.01	0.060152
0.001	0.0027405
0.0001	0.00062997
0.00001	0.00038689
0.000001	0.000052316

When performing net weight measurements, analysts must follow the following minimum weight thresholds requirements. These minimum values ensure (95% level of confidence) that the relative uncertainty associated with the balance used is no greater than 1% of the weight measurement recorded. Minimum weight thresholds are applicable to each individual net weight measurement, not to the total net weight of the exhibit. Minimum weight thresholds do not apply to tared containers (paper, weighing boats, original or substitute packaging, glassware, etc.).

NOTE: When doing container extrapolation, minimum weight thresholds are applied to the individual container weights.

Minimum weight thresholds are obtained using Equation 2, where  $k = 2$  corresponds to a 95% level of confidence,  $U_{rel}$  is the relative uncertainty requirement of 1%, and *Median SD* is the system-wide median standard deviation calculated from all (low and high-load) performance verification measurements for each balance type.

$$\text{Min. weight} = \frac{k}{U_{rel}} (\text{Median SD}) \quad \text{Equation 2}$$

<u>Readability (g):</u>	<u>Minimum Weight (g):</u>
0.1	20.0
0.01	2.50
0.001	0.250
0.0001	0.0300
0.00001	0.01500
0.000001	0.001000

The traceability of weight measurements is established through the use of balances calibrated to traceable reference standards. Measurement assurance is provided by monthly balance performance verification procedures per 1-6.3.1. The DEA property inventory number of the balance is documented in LIMS. All net weight, volume, unit count, and uncertainty calculations are determined using the DEA Uncertainty Calculator within the LIMS *Net Weight* test, and a copy of the completed worksheet is included in the case file.

### 1.1.1 Determination of UME for Direct Weight Cases

When the net weight of an exhibit is obtained by direct measurement(s), the uncertainty associated with the total net weight ( $U_{NW}$ ) is obtained by multiplying the combined weighing uncertainty ( $u_w$ ) by a coverage factor ( $k$ ) corresponding to a 95% level of confidence (Equation 3). As a conservative estimate, all weighing events are assumed and treated as static processes<sup>2</sup> and correlated measurements; therefore, all combined net weight uncertainties are calculated by

<sup>2</sup> A static weighing process involves two separate weighing events, the weighing of the vessel by itself and the weighing of the vessel with material.

linear addition of the standard uncertainties associated with each individual weighing event (Equation 4).

$$U_{NW} = u_w \times k \quad \text{Equation 3}$$

where,

$$u_w = u_1 + u_2 + u_3 + \dots + u_n \quad \text{Equation 4}$$

$u_n$  = individual uncertainty ( $u_{mass}$ ) of weighing event  $n$

$k = 2$ , for a 95% level of confidence

The total net weight and uncertainty of the exhibit is:

$$\text{Net weight} \pm U_{NW} = \text{Net weight} \pm (u_w \times k)$$

### 1.1.2 Determination of UME for Extrapolation Cases

When the total net weight of an exhibit is obtained by extrapolation, two sources of uncertainty are considered, the uncertainty associated with the calculated average weight per unit ( $u_{avg}$ ) and the uncertainty associated with the balance used ( $u_{mass}$ ). The uncertainty contribution from the balance is obtained from system-wide monthly performance verification data and the uncertainty associated with the calculated average weight per unit is determined using Equation 5.

$$u_{avg} = \frac{s}{\sqrt{n}} \quad \text{Equation 5}$$

where,

$s$  = sample standard deviation from individual weight measurements

$n$  = number of units individually weighed

The average net weight and combined uncertainty per unit ( $u_{NW}$ ) are:

$$\text{Average NW} \pm u_{NW} \quad \text{where,} \quad u_{NW} = \sqrt{u_{mass}^2 + u_{avg}^2}$$

The total extrapolated net weight and uncertainty for the exhibit are:

$$\text{Net weight} \pm U_{NW} = (\text{total \# units}) (\text{Avg. NW} \pm u_{NW} \cdot t_{95\%})$$

(coverage factor used is  $t_{95\%} = 2.306$ , corresponding to the Student's- $t$  value for 8 degrees of freedom at the 95% level of confidence).

Acceptance criteria: For extrapolation cases, exhibit units are considered uniform (based on contents or container) if the RSD obtained from the nine individual measurements performed is 10% or less. Final uncertainty values associated with net weight determinations are acceptable if the calculated relative uncertainty (U/NW) is 25% or less. If higher RSD or relative uncertainty values are obtained, alternative approaches to net weight determination should be pursued. For example, use of a higher precision balance, extrapolation by container instead of contents, weighing of units by groups of higher uniformity, etc. Analysts and supervisory personnel should evaluate these situations on a case by case basis.

NOTE: For situations not covered above (non-uniform units, two-layer liquids, mixtures of solids and liquids, etc.), net weight, volume, and total unit count determinations are left to the discretion of the analyst and supervisory personnel. REDACTED

## 1.2 Purity

The uncertainty associated with purity determinations is assessed by considering four contributing factors: reproducibility, accuracy (bias), sample preparation, and reference materials. Together, these factors take into consideration the most significant components associated with the total estimated uncertainty.

For all quantitative analyses, regardless of analyte, laboratory, matrix or analytical methodology used, the total expanded uncertainty ( $U$ ) associated with the final purity result is obtained using Equation 6:

$$U = \%P \cdot u_c \cdot k_{95\%} \quad \text{Equation 6}$$

where,

$$u_c = \sqrt{u_R^2 + u_{Acc}^2 + u_{PTP\ Spl}^2 + u_{RM}^2}$$

and,

$\%P$  = empirically-determined purity of the analyte

$u_c$  = combined relative uncertainty

$k_{95\%}$  = coverage factor for a 95% level of confidence ( $k = 2$ )

$u_R$  = concentration-dependent relative uncertainty associated with the laboratory system's reproducibility (or coefficient of variation)

$u_{acc}$  = concentration-dependent relative uncertainty associated with the laboratory system's accuracy (bias)

$u_{PTP\ Spl}$  = relative uncertainty associated with the gravimetric preparation of the Proficiency Testing Program (PTP) samples generated by SFL1.

$$u_{PTP\ Spl} = 0.0002131$$

$u_{RM}$  = relative uncertainty associated with the purity of the reference materials used in the laboratories for preparation of the calibrant solutions.

$$u_{RM} = 0.005774$$

### 1.2.1 Reproducibility ( $u_R$ )

System-wide laboratory proficiency testing (PTP) data are used to evaluate the reproducibility of DEA's quantitative processes. Evaluation of over five years (2014-2018) of PTP results indicates that the relative standard deviation (RSD) obtained for quantitative analyses can be estimated by a natural log function of concentration (% purity), with higher RSD values observed as the concentration of the analyte decreases. This behavior is similar to that previously characterized and documented by Horwitz and collaborators<sup>34</sup> during the evaluation of more than 100 years of inter-laboratory studies. Horwitz observed that an approximately 2-fold increase in RSD occurs for each 100-fold decrease in analyte concentration. These studies also demonstrated that the RSD associated with purity determinations is independent of analyte, matrix, or analytical technique used.

Figure 1 shows results from DEA PTP samples analyzed during the years 2014-2018. Each data point represents the RSD obtained from multiple analyses of one PTP within the DEA laboratory system. Figure 1 also illustrates the natural log function (solid line) describing the dependence of RSD on concentration, mathematically illustrated by Equation 7.

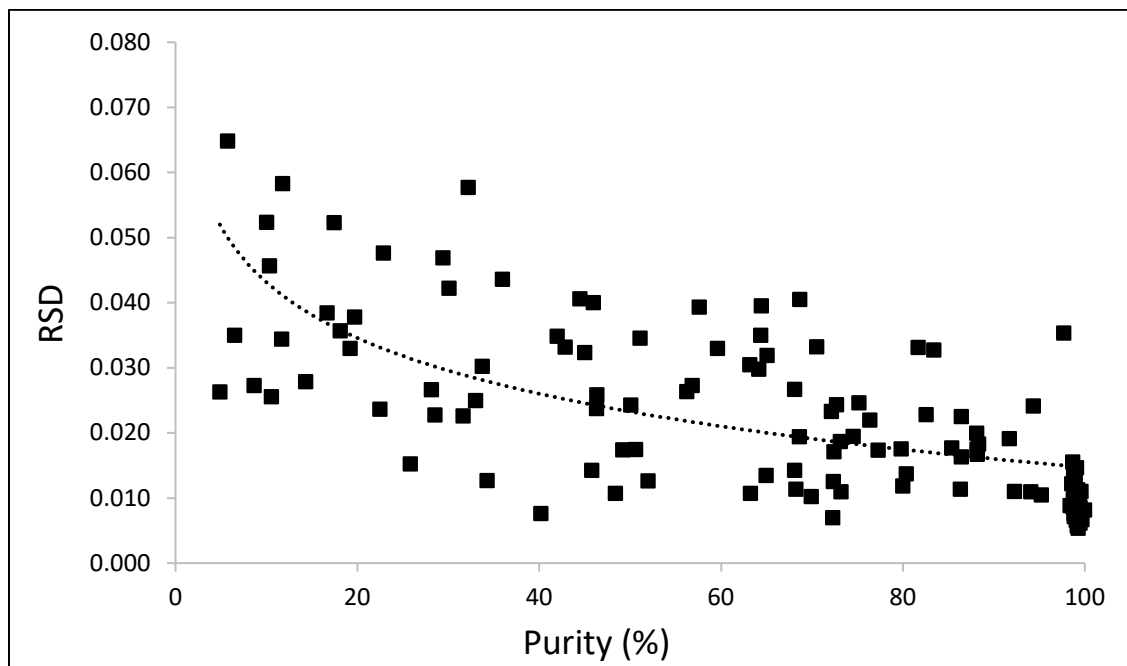
$$RSD = 0.0716 - 0.012\ln(\%P) \qquad \text{Equation 7}$$

---

<sup>3</sup> Horwitz W, Kamps LR, Boyer KW. Quality assurance in the analysis of foods for trace components. J. Assoc. Off. Anal. Chem. 1980; 63(6):1344-1354.

<sup>4</sup> Boyer KW, Horwitz W, Albert R. Interlaboratory variability in trace element analysis. Anal. Chem. 1985; 57:454-459.





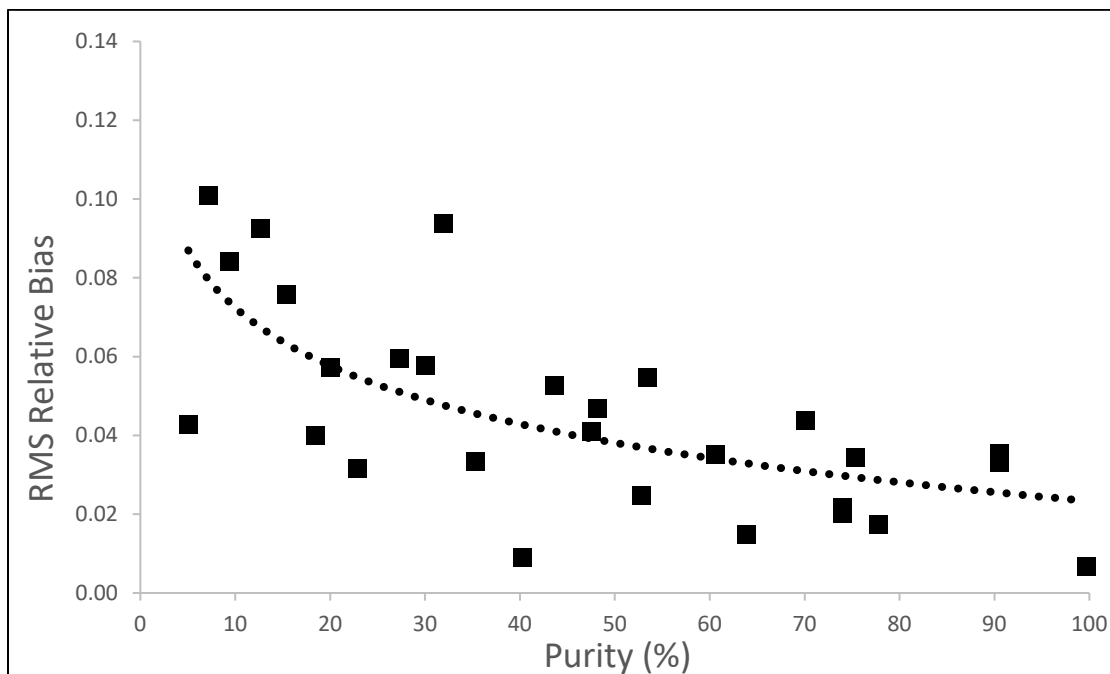
**Figure 1:** Dependence of PTP RSD values on purity

$u_R$  represents the variability of purity results across laboratories and incorporates contributing factors such as different analytes, purity levels, laboratories, analysts, methods, environments, sample preparations, instruments, balances, volumetric glassware, reagents, and consumables.

### 1.2.2 Accuracy ( $u_{Acc}$ )

The DEA laboratory system-wide accuracy (bias) is evaluated by using PTP samples prepared by SFL1 during 2014-2018. The evaluation of 28 samples of known composition also indicates a concentration-dependent behavior that can be estimated by a natural log function. In Figure 2, each data point represents the root-mean-square (RMS) relative bias calculated from analysis of each of the 28 PTP samples prepared and distributed by SFL1. Figure 2 also includes the natural log function estimating the dependence of RMS relative bias on concentration. Equation 8 mathematically illustrates the  $u_{acc}$  component.

$$u_{acc} = 0.1215 - 0.021\ln(\%P) \quad \text{Equation 8}$$



**Figure 2:** Dependence of RMS relative bias on concentration for PTP samples prepared by SFL1 (2014-2018)

$u_{acc}$  represents the accuracy variability across laboratories and incorporates all the contributing factors such as different analytes, purity levels, laboratories, analysts, methodology, environments, sample preparations, instruments, balances, volumetric glassware, reagents, and consumables.

### 1.2.3 PTP Sample Preparation ( $u_{PTP\ spl}$ )

For each PTP sample generated by SFL1, the uncertainty associated with the preparation is calculated based on the number of weighing events involved and the balance type used.  $u_{PTP\ spl}$  represents the overall average relative combined uncertainty for all SFL1-prepared samples. This component also provides traceability to the reference standard weights used to calibrate the SFL1 balances. For the current data period, the value used is  $u_{PTP\ spl} = 0.0002131$ .

### 1.2.4 Reference Material ( $u_{RM}$ )

$u_{RM}$  represents the relative uncertainty associated with the SFL1-produced reference materials and used by the field laboratories to prepare calibrant solutions (calibration curves). For the current data period, the value used is  $u_{RM} = 0.005774$ , obtained from the purity requirement for reference materials (> 98%) and assuming a uniform (square) distribution of values.

### 1.3 Amount of Pure Substance

The uncertainty associated with the amount of pure substance is calculated by combining the standard relative uncertainties associated with net weight and purity, using the root-sum-of-square (RSS) method for uncorrelated quantities. The total amount of pure substance (APS) and uncertainty is obtained as follows:

$$APS \pm U_{APS} = APS \pm (APS) \left( u'_{APS} \right) (k) = APS \pm (APS) \left( \sqrt{\left( u'_{NW} \right)^2 + \left( u'_p \right)^2} \right) (k)$$

where,

$u'_{APS}$ ,  $u'_{NW}$ , and  $u'_p$  are the relative uncertainties associated with amount of pure substance, net weight, and purity, such that

$$u'_{APS} = \left( \frac{u_{APS}}{APS} \right) = \left( \sqrt{\left( u'_{NW} \right)^2 + \left( u'_p \right)^2} \right) \text{ and } u'_{NW} = \left( \frac{u_{NW}}{NW} \right) \text{ and } u'_p = \left( \frac{u_p}{p} \right)$$

The final uncertainties associated with purity and amount of pure substance determinations are automatically calculated within LIMS as part of the *Summary of Findings* test.

## **Appendix 2G – Instructions for Completing LIMS Tests**

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## **1 Gross Weight Test**

- Weigh the sealed evidence as received from the vault using the balance software.
- Ensure the *Gross Weight (Actual)* finding is populated correctly.
  - For sub-exhibits, the *Gross Weight* test is added only to the first sub-exhibit.
- Obtain a witness for the *Weight Discrepancy* finding, if needed.
- Document the balance used in the *Equipment* tab.

## **2 Description of Evidence Test**

- For sub-exhibits, the *Description of Evidence* test is added only to the first sub-exhibit.
- Record the condition of the seals as received in the *Seals* finding.
  - Obtain a witness in the *Seal Witness* finding, if the seals are not intact.
- Enter the date the evidence was opened in the *Date Opened* finding.
  - If the evidence was opened more than once, annotate the *Remarks* finding with each opening date.
- In the *Description* finding, provide a description of the physical evidence, including containers, markings, drug gross form (crystalline, powder, etc.), and other information in sufficient detail that a reader can visualize the evidence.
  - If the description of the evidence is too long for the provided space, a PDF document shall be attached to this test and the *Description* finding should include the following “See attached document for evidence description.”
- Select “Yes/No” in the *Consistent with Paperwork* finding as appropriate.
- Obtain a witness to any discrepancy.
  - A supervisor or another FC enters one’s username and password to document the witnessing of the *Description* discrepancy.

## **3 Description of Exhibit and Sampling Test**

- Add the *Description of Exhibit and Sampling* test to all sub-exhibits.
  - Describe the packaging in the first sub-exhibit when the sub-exhibits are submitted in one container.
  - Set the *Package Type* finding to “*Described in first exhibit split*” for all of the remaining sub-exhibits.

- Enter the number of innermost packages containing the suspected controlled substance in the *Number of Packages* finding.
- Enter the total number of units in the exhibit in the *Number of Units* finding.
  - For commingled residue exhibits, enter the *Number of Units* as 1.
- Select the appropriate descriptions for the innermost packaging and the contents of the exhibit in the *Package Type* finding.
  - Select “*Other*” when the exhibit contains multiple package types or when the package type is not listed.
    - The packaging shall be described in the *Remarks* finding.
    - Include a statement describing the packaging in the *Exhibit Details* section on the DEA-113.
- Set the *Logo/Impression* finding to “Yes” if a submission to *Operation Fountainhead* is made.
- Select the appropriate form of the material to be analyzed in the *Gross Form* finding.
  - Select “*Other*” when the exhibit contains different forms or when the form is not listed.
    - The gross form shall be described in the *Remarks* section.
    - Include a statement describing the form(s) in the *Exhibit Details* section on the DEA-113.
- Select “*Dry*”, “*Moist*” or “*N/A*”, as applicable.
- Set the *Exemplar* finding to “Yes” if the sample is an exemplar exhibit.
- Enter the number of units analyzed in the *Number of Units Tested* finding.
- Provide details on how the exhibit was sampled in the *Sampling Procedure* finding. Describe the sampling procedure used to select the units for identification, including the random sampling technique (if used), and the tests used during pre-composite testing. Document the composite formation and particle size reduction procedure(s)(if used), including the sieve size (or particle size).
- Obtain supervisory approval for deviations from the evidence sampling plan (ESP).
  - The supervisor electronically approves the deviation in the *Deviation Approved By* finding before the analysis is completed.
  - Documents the reason for the deviation in the *Reason for Deviation* finding.
- All pictures of the exhibit not related to another specific LIMS test shall be attached to the *Image* finding.

#### **4 Net Weight Test**

- Select “Yes/No” in the *Residue* finding, as appropriate.
- Select the type of weighing performed in the *Type of Weighing* finding.
- Obtain the net weight of the exhibit using the balance software.
- For liquids and solutions: use the balance software to determine the density of the composite and calculate the total volume of the exhibit.
- For solid dosage forms: determine the total number of units by counting or by extrapolation using the balance software.
- Describe the item(s) used to obtain the tare weight, if different from original packaging (e.g., substitute packaging, weigh boat, etc.) in the *Remarks* finding.
- Record weights and the number of units (e.g., *volume, number of dosages units, etc.*) with sufficient accuracy to meet the requirements specified in 2-3.4 and 2-11.4 (DO NOT round up a number).
- Ensure the net weight and the net weight uncertainty populate the *Net Weight* test fields correctly.
- Document the use of the *Legacy Calculator* and obtain supervisory approval of the deviation.
- Document the balance used in the *Equipment* tab.

#### **5 Net Weight (Sub-Group) Test**

- Select the type of weighing performed in the *Type of Weighing* finding.

NOTE: This test will automatically populate after the completion of all Sub-Group balance task(s).

- Describe the item(s) used to obtain the tare weight, if different from the original packaging (e.g., substitute packaging, weigh boat, etc.) in the *Remarks* finding.
- Ensure the net weight and the net weight uncertainty populate the *Net Weight* test fields correctly.
- Document the use of the *Legacy Calculator* and obtain supervisory approval of the deviation.
- Document the balance used in the *Equipment* tab.

#### **6 Net Weight (Exemplar) Test**

- Use this test only for exemplar exhibits.
- Select the type of weighing performed in the *Type of Weighing* finding.



- Use the *Exemplar Different A* balance method for exhibits composed of one representative and multiple core-type samples.
- Use the *Exemplar Same Container* balance method for all other types of exhibits.
- Enter the total number of units in the *Number of Units* finding.
- Enter the net weight in the *Total Net Weight of Exhibit* finding.
- Describe the item(s) used to obtain the tare weight, if different from the original packaging (e.g., substitute packaging, weigh boat, etc.) in the *Remarks* finding.
- Document the balance used in the *Equipment* tab.

## **7 Composite Weight Test**

- Use this test as needed.
- Set the *Reserve Composite Weighed* finding to “Yes” or “No”, as applicable.
- Enter the initial composite weight and reserve composite weight in the appropriate findings.
- Describe the item(s) used to obtain the tare weight (e.g., packaging, substitute packaging, etc.) in the *Remarks* finding.
- Document the balance used in the *Equipment* tab.

## **8 Additional Evidence Unit Test**

- Add this test to all evidence containers except the first evidence container when there are multiple evidence containers (i.e., two PSEEs, five tape sealed cardboard boxes, etc.).
- Add the phrase “*All tests listed in Unit 1*” in the *Remarks* finding.

## **9 No Analysis Performed Test**

- Enter “*No Analysis Performed*” in the *Results* finding.
- Enter the authorization for not performing the analysis (e.g., No analysis per S/A John Doe, etc.) into the *Reason* finding.

## **10 Logo Identification**

- This test is used for information only and cannot be used to fulfill the minimum identification requirements of two tests/two portions.
- Record the logo identification information (i.e., 5 mg Oxycodone, 650 mg Acetaminophen) in the *Logo ID* finding.

- Record the source used to make the logo identification in the *Logo ID Source* finding.
  - If the source of the logo identification was the manufacture's website, record the internet address of the web page used to make the identification in the *Manufacturer Website* finding.
  - If the source is not listed in the *Logo ID Source* finding, select "Other" and record the source used in the *Remarks* finding.
- If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- Make an annotation as to which units were tested in the *Remarks* finding.
  - The annotation must be self-documenting so that it is clear to which units the results apply.

NOTE: The annotation "x29" is not self-documenting and therefore may not be used in the *Remarks* finding to describe which units the results apply.

#### **11 pH Test**

- Record the method used to make the pH measurement in the *Method of pH Measurement* finding.
- Record the pH measured in the *Measured pH* finding.

#### **12 Solubility/Miscibility Test**

- Select either the *Solubility* or *Miscibility* finding.
- Record the solvent used in the *Solvent* finding.
- Select the appropriate value for the *Miscibility Result* finding, if tested.
- Select the appropriate value for the *Solubility Result* finding, if tested.
- If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., "Result applies to Units 1–29").

NOTE: The annotation "x29" is not self-documenting and therefore may not be used in the *Remarks* finding.

#### **13 Watesmo Paper Record Test**

- Select the appropriate value in the *Color Observed* finding.

- If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- Annotates which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

#### **14 Macro/Microscopic Examination of Plant Material**

- Record the macroscopic description of the plant material in the *Macroscopic Observation* finding.
  - Select “Other” for the *Macroscopic Observations* finding to enter an explanation in the *Remarks* finding.
- Enter the magnification used to examine the plant material in the *Magnification* finding.
- Record all observations made while performing the microscopic examination in the *Microscopic Observations* finding.
  - Select “Other” for the *Microscopic Observations* finding to enter an explanation in the *Remarks* finding.
- Document the microscope(s) used in the *Equipment* tab.
- If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

#### **15 Microscopic Examination Test**

- Use this test to document characteristics of the exhibit (i.e., cube shaped crystals) and foreign material (i.e., white powder, etc.) found on plant material.
- Enter the magnification used to examine the material in the *Magnification* finding.
- Record all observations made while performing the microscopic examination in the *Microscopic Observations* finding.
- Document the microscope(s) used in the *Equipment* tab.
- If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).

- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

## 16 Color Tests

- Uses this procedure for all color tests.
- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
  - Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
  - Select the appropriate color observed from the values in the *Color* finding.
  - If no matching color exists in the drop-down menu, select “Other” and record the color observed in the *Remarks* finding.
- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

## 17 Crystal Tests

- Use this procedure for the following tests: *Gold Chloride Crystal Test*, *Platinic Chloride Crystal Test*, *Barium Nitrate Crystal Test*, *Copper Sulfate Crystal Test*, and *Silver Nitrate Crystal Test*.
- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
  - Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
  - Enter the observations in the *Crystals Observed* finding.

- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

## 18 Thin Layer Chromatography Test

- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
  - Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
- Select the appropriate TLC plate, sample solvent, developing solvent system, and visualizing reagent used in the *Plate*, *Solvent*, *Solvent System*, and *Visualizing Agent* findings, respectively.
  - If no matching value exists in the drop-down menu in the *Plate*, *Solvent*, and/or *Solvent System* findings, select “Other” and record the *Plate*, *Solvent*, and/or *Solvent System* used in the *Remarks* finding.
- Record the name(s) of the compound(s), reference materials, and retention factor(s) into the *Results* finding.
- Annotate which units were tested and give the same results in the *Remarks* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used in the *Remarks* finding.

## 19 Chromatographic Tests

- Use this procedure for the following tests: *CE Analysis*, *HPLC Analysis*, and *GC Analysis*.
- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
  - Indicate if the sample was weighed in the *Sample Weighed* finding.

- Enter the appropriate values into the *Sample Prep* findings.
- Record the solvent used to dissolve the sample (e.g.,  $\text{CHCl}_3$ , sample dissolved in  $\text{H}_2\text{O}$  and basified with 1.0 N  $\text{NaOH}_{(\text{aq})}$  and extracted into  $\text{CH}_2\text{Cl}_2$ , etc.) in the *Solvent* finding.
- Record the name(s) of the compound(s), reference materials, retention/migration time(s), and corresponding area counts into the *Chromatographic Results* finding for each sample.
- Document the instrument(s) and balance(s) used in the *Equipment* tab.

## 20 Hyphenated Tests

- Use this procedure for the following tests: *GC-IRD Analysis*, *GC-MS Analysis*, and *LC-MS Analysis*.
- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail”.
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
  - Indicate if the sample was weighed in the *Sample Weighed* finding.
  - Enter the appropriate values into the *Sample Prep* findings.
- Record the solvent used to dissolve the sample (e.g.,  $\text{CHCl}_3$ , Sample dissolved in  $\text{H}_2\text{O}$  and basified with 1.0 N  $\text{NaOH}_{(\text{aq})}$  and extracted into  $\text{CH}_2\text{Cl}_2$ , etc.) in the *Solvent* finding.
- Set the *Retention Time Matching* finding to “No” for spectral results only.
  - Add the name(s) of the compound(s) identified to the *Spectral Result* finding.
  - If the separation test (retention times) is used to fulfill the identification requirements, the results must be documented.
- Set the *Retention Time* finding to “Yes” for the instrument to perform retention time calculations.
  - Record the name(s) of the compound(s), reference material, retention time(s), and corresponding area counts in the *Chromatographic Results* finding.
- Document the instrument(s) and balance(s) used in the *Equipment* tab.

## 21 NMR Test

- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail”.
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
  - Indicate if the sample was weighed in the *Sample Weighed* finding.
  - Enter the sample weight and dilution volume into the *Sample Weight* and *Final Dilution Volume* findings, respectively.
- Select the solvent used for the *Solvent* finding.
  - If no matching solvent exists in the drop-down menu, select “Other” and record the solvent used in the *Remarks* finding.
- Enter the name(s) of the compound(s) identified into the *Results* finding.
- Document the instrument(s) and balance(s) used in the *Equipment* tab.

## 22 Vibrational Spectroscopy Tests

- Use this procedure for the following tests: *FTIR Analysis* and *Raman Analysis*.
- Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
  - Enter the sample preparation used (e.g., Direct, CHCl<sub>3</sub> Solubles, Acetone Insolubles/CHCl<sub>3</sub> Solubles, etc.) in *Sample Prep* finding.
- Enter the name(s) of the compound(s) identified into the *Results* finding.
- Indicate the instrument(s) used in the *Equipment* tab.

## **23 UV-Vis Test**

- Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
- Enter the name(s) of the compound(s) identified into the *Results* finding.
- Document the instrument(s) used in the *Equipment* tab.

## **24 IMS Test**

- Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Record the result for each sample that corresponds to a specific negative control in the same run using subsequent sets.
- Set the *Verification Test* to “Yes” if the sample is a verification sample.
- Record the *Verification Test Result*: “Pass” or “Fail.”
- Record the name of the compound(s) identified in the *Results* finding.
- Document the instrument(s) used in the *Equipment* tab.

## **25 Polarimetry Test**

- Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
  - Select the appropriate value for the *Negative Control Type* finding.
  - Record the *Negative Control Result* finding as “Pass” or “Fail.”
- Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
- Enter the observed optical rotation in the *Observed Rotation* finding.
- Document the instrument(s) used in the *Equipment* tab.



**26 Deviation Test**

- Describe the deviation in the *Description of Deviation* finding.
- Documents approval of the deviation in the *Deviation Approved By* finding.

**27 Quantitation Test**

- Select “*Sample*,” “*Standard*,” “*Check*,” or “*Blank*” in the *Type* finding.
- Indicate the method name/number and technique used (i.e., DEA101/Gas Chromatography, COC-LC/Liquid Chromatography, etc.).
- Provide details of any modification to the validated method in the *Remarks* finding.
  - Obtains supervisory approval in the *Deviation* test.
- Documents the type of dilution in the *Dilution Technique* finding, if applicable.
  - For *Volumetric*, *Gravimetric*, and *Volumetric/Gravimetric* dilutions, complete all applicable sample preparation findings.
- Enter the appropriate amount in the *Sample Amount (Instrument)* finding.
  - For the reference material, use the purity-corrected concentration or weight of the calibrant.
  - For a QC solution, use the total solution concentration or weight to assess the QC reference purity value directly; or use the purity-corrected concentration to assess the QC reference purity value normalized to 100%.
- Enter the appropriate factor in the *Dilution Factor* finding.
- Document the calibrant(s) and QC solution preparations in the *Remarks* finding or as an attachment. Include the following information:
  - Name, salt form, and lot number/identifier of the reference material or QC sample used.
  - Weight, volume, dilution, final concentrations, and preparations date(s).
- Document the instrument(s) and balance(s) used in the *Equipment* tab.
- Use the following conversion factors as the multiplier when salt form corrections are required:

	Salt Conversion	Factor
<i>Cocaine</i>	HCl → Base	0.8929
	Base → HCl	1.1200
<i>Heroin</i>	HCl → Base	0.8714
	Base → HCl	1.1475
<i>Methamphetamine</i>	HCl → Base	0.8034
	Base → HCl	1.2446
<i>Amphetamine</i>	HCl → SO <sub>4</sub>	1.0737
	HCl → Base	0.7879
	SO <sub>4</sub> → HCl	0.9313
	SO <sub>4</sub> → Base	0.7338
	Base → HCl	1.2692
	Base → SO <sub>4</sub>	1.3628
<i>PCP</i>	HCl → Base	0.8696
	Base → HCl	1.1500
<i>BZP</i>	HCl → diHCl	1.1712
	HCl → Base	0.8288
	diHCl → HCl	0.8539
	diHCl → Base	0.7077
	Base → HCl	1.2065
	Base → diHCl	1.4130

Final factors are rounded values.

## 28 Other Notes Test

- This test is used to record any relevant procedures, testing, or observations not captured in other LIMS tests.

## 29 REDACTED

- REDACTED
  - REDACTED
- REDACTED
- REDACTED
- REDACTED

## 30 Exemplar Weight Removed Test

- Add this test to the parent exhibit.

- Enter the total amount of material removed for *REDACTED*.
- Document the balance used in the *Equipment* tab.

**31 REDACTED**

- REDACTED
- REDACTED
- REDACTED
- REDACTED

**32 REDACTED**

- REDACTED
- REDACTED
- REDACTED

**33 REDACTED**

- REDACTED
- REDACTED
- REDACTED
- REDACTED

**34 REDACTED**

- REDACTED
- REDACTED
- REDACTED

**35 REDACTED**

- REDACTED
- REDACTED

- REDACTED
- REDACTED
- REDACTED.
- REDACTED

**36 Gross Weight – REDACTED/Latent Print (REDACTED/LP) Test**

- Add this test to an exhibit that qualifies for a REDACTED sampling, Latent Print examination, or defense analysis sample (DFA).
- Indicate the weight of the sealed *REDACTED*, *Latent Print*, or *DFA* evidence.
- Document the balance used in the *Equipment* tab.

**37 Reserve Weight Test**

- Select “Yes” or “No” in the *Residue* finding.
- In the *Type of Calculation* finding, select: “*Calculate Volume*”, “*Calculate Dosage Units*” or “*No Calculation*” based on the gross form of the exhibit (liquid, dosage units, or all others, respectively).
- Enter the final reserve weight of exhibit in the *Reserve Weight* finding.
  - Reserve weight is reported using the same units as the reported net weight.
  - For bulk exhibits, the reserve weight is the entire remaining amount including threshold and bulk portions.
- For liquid exhibits, enter the total reserve volume in the *Reserve Volume* finding.
- For dosage units, enter total reserve unit count in the *Reserve Dosage Units* finding.
- Describe the item(s) used to obtain the tare weight, if different from original packaging (e.g., substitute packaging, weigh boat, etc.) in the *Remarks* finding.
- Document the balance used in the *Equipment* tab.

**38 Reserve Net Weight (Exemplar) Test**

- Select the type of weighing performed in the *Type of Weighing* finding.
  - Use the *Reserve Exemplar Different A* balance method for exhibits composed of one representative and multiple core-type samples.

- Use the *Reserve Exemplar Same Container* balance method for all other types of exhibits.
- Enter the total number of units in the *Number of Units* finding.
- Enter the final reserve weight in the *Total Reserve Net Weight of Exhibit* finding.
  - Reserve weight is reported using the same units as the reported net weight.
- Describe the item(s) used to obtain the tare weight, if different from original packaging (e.g., substitute packaging, weigh boat, etc.) in the *Remarks* finding.
- Document the balance used in the *Equipment* tab.

### **39 Reserve Weight (Direct Bulk) Test**

- This test is used to document the amount separated for threshold and bulk, when the *Reserve Weight (Bulk)* test is not used.
- Use this test in conjunction with the *Reserve Weight* test.
  - For DHS bulk exhibits in which the threshold and bulk portions are not separated, the bulk reserve weights tests should not be utilized.
- Enter the total reserve weight in the *Total Reserve Net Weight* finding.
- Enter the amount of the exhibit to be retained in the *Threshold Weight* finding.
- Enter the amount of the exhibit to be destroyed in the *Bulk Weight* finding.

### **40 Reserve Weight (Bulk) Test**

- This test may be used to obtain the reserve weight of a bulk exhibit when the net weight was obtained by extrapolation.
- Use this test in conjunction with the *Net Weight*, *Composite Weight*, and *Exemplar Weight Removed* tests.
  - For DHS bulk exhibits in which the threshold and bulk portions are not separated, the bulk reserve weights tests should not be utilized.
- In the *Type of Calculation* finding, select “*Calculate Volume*,” “*Calculate Dosage Units*,” or “*No Calculation*,” based on the gross form of the exhibit (liquid, dosage units, or all others, respectively).
- Indicates the initial net weight, amount of composite used, REDACTED in the corresponding findings.
- Indicates the number of units placed in the threshold container for the *Total Units in Threshold* finding.

- Populates the *Average Weight per Container* finding from the *Uncertainty Calculator*.
- Calculates the *Threshold Weight* and *Bulk Weight* findings.

#### **41 Description of Reserve Evidence Test**

- Describe all reserve evidence items in the *Description* finding.
  - For sub-exhibits, add this test to the first unit and include a description of each sub-exhibit.
  - Document any packaging changes.

NOTE: If the description of the evidence is too long for the provided space, a PDF document shall be attached to this test and the *Description* finding should include the following “See attached document for evidence description.”

- Record the date the container was sealed in the *Date Sealed* finding.
- If applicable, document each instance the container(s) was reopened and resealed in the *Remarks* finding.

#### **42 Gross Weight After Analysis Test**

- Record the weight of the sealed evidence after analysis.
  - For sub-exhibits, the *Gross Weight After Analysis* test is added only to the first sub-exhibit.
- Indicates the balance used in the *Equipment* tab.

#### **43 Supervisory Approval Test**

- In the *Description of Action Taken* finding, describe the scenario or action requiring supervisory approval.
- Documents approval obtained in the *Action Approved* finding, before the analysis is completed.

#### **44 SLite Solution Tests (SFL1 only)**

- For sub-exhibit scenarios, add this test to each sub-exhibit.
- For the *Gross Weight* findings of sub-exhibits (e.g., X.02, X.03, etc.), enter “N/A”.
- For residue scenarios, net and reserve weights that are residue, enter “0.001g” in the *Net Weight* and *Reserve Weight* findings.
- When the main drug salt form is not determined and the quantitation value is reported “*calculated as*”, enter the primary drug with no salt form as the component in the *Quantitation* finding. (Place a note in the *Remarks* finding that it was “*calculated as*”.)

- When no quantitation is performed, complete the *Quantitation* finding with “N/A”.
- Report quantitative analysis results as directed in Appendix 2F-45.
- If no analysis is performed, add the appropriate test and select “No Analysis Performed” as the *Qualitative* finding.
- Complete all other findings with “N/A”.
- REDACTED

#### **44.1 Enforcement Exhibit Test**

- Indicate the date the exhibit was received in the *Date Received* finding.
- Enter the weight of the sealed evidence in the *Gross Weight* finding.
- Enter the net weight of the exhibit or sub-exhibit in the *Net Weight* finding.
- Indicate the number of innermost packages containing the suspected controlled substance in the *Number of Packages* finding.
- Enter appropriate descriptions for the innermost packaging and the contents of the exhibit in the *Package Type* and *Package Color* findings.

- Enter the reserve weight of the exhibit or sub-exhibit in the *Reserve Weight* finding.
- Enter the name(s) of the compound(s) identified into the *Qualitative Results* finding.
- Enter the name(s) of the compounds(s) quantitated and their corresponding purity in the *Quantitative Results* finding.
- Enter all weights as they are to appear on the final report.
- Indicate the date the final report was submitted for approval in the *Date Completed* finding.

#### **44.2 Enforcement Exhibit Test**

- REDACTED

#### **44.3 REDACTED**

- REDACTED
- REDACTED
- REDACTED
- REDACTED
- REDACTED
- REDACTED
- REDACTED
- REDACTED
- REDACTED

#### **45 Summary of Findings Test**

- This test is used to report conclusions from the exhibit's analysis.
- Add this test to the first sub-exhibit only, but includes the conclusions for all sub-exhibits.
- Include all information necessary to complete the DEA-113 including: *Gross Weight, Substance(s) Identified, Net Weight, Substance Purity, Amount Pure Substance, Associated Uncertainties, and Reserve Weight.*



- *Substance(s) Identified:* Annotate all controlled, listed, and non-controlled substance(s) identified. Include isomer and salt form, if identified.

NOTE: The order in which the substances identified are added to the *Summary of Findings* test determines the reporting order on the DEA-113. Refer to 2-11.4 for reporting order.

- *Substance Purity:* Report purity and UME as percent.
- *Amount Pure Substance (APS):* The APS is the product of the reported (truncated) net weight and the reported (truncated) purity.

NOTE 1: Truncate and report APS in the same units and to the same significance as the net weight.

NOTE 2: The uncertainty associated with APS is a rounded value reported to the same significance as the APS.

- *Reserve Weight:* Report in the same units as the net weight.

REDACTED

REDACTED

**TABLE 1**

Units to be randomly selected for qualitative analysis, no negative results expected. Allows an inference on at least 90% of the population at the 95% (or higher) level of confidence when no negative results are observed.

Total # of Units	Units to be Selected	Total # of Units	Units to be Selected
10-12	9	38-46	20
13	10	47-48	21
14	11	49-58	22
15-16	12	59-77	23
17	13	78-88	24
18	14	89-118	25
19-24	15	119-178	26
25-26	16	179-298	27
27	17	299-1000	28
28-35	18	> 1000	29
36-37	19		

**TABLE 2**

Minimum test sample amounts (in mg) required for preparation of quantitation solutions for exhibits with net weight equal or greater than 100 mg.

**Powders, Crystalline Materials and Solid Dosage Forms (NW ≥ 100 mg)**

**Test amounts (mg)<sup>a</sup> for composites ground, mixed, and sieved to 850 µm (20-mesh): 425 µm (40-mesh) and 250 µm (60-mesh):**

<b>Expected Purity (%):</b>	<b>850 µm (20-mesh):</b>	<b>425 µm (40-mesh):</b>	<b>250 µm (60-mesh):</b>
0.5	38249	4782	974
1	19057	2383	485
2	9461	1183	241
3	6262	783	160
4	4662	583	119
5	3702	463	95
10	1780	223	46
15	1137	143	29
20	814	102	21
25	619	78	16
30	489	62	13
35	394	50	11
40	323	41	>10 <sup>b</sup>
45	267	34	>10 <sup>b</sup>
50	221	28	>10 <sup>b</sup>
55	183	23	>10 <sup>b</sup>
60	151	19	>10 <sup>b</sup>
65	124	16	>10 <sup>b</sup>
70	100	13	>10 <sup>b</sup>
75	79	>10 <sup>b</sup>	>10 <sup>b</sup>
80	60	>10 <sup>b</sup>	>10 <sup>b</sup>

<b>Expected Purity (%):</b>	<b>850 <math>\mu\text{m}</math> (20-mesh):</b>	<b>425 <math>\mu\text{m}</math> (40-mesh):</b>	<b>250 <math>\mu\text{m}</math> (60-mesh):</b>
85	43	>10 <sup>b</sup>	>10 <sup>b</sup>
90	28	>10 <sup>b</sup>	>10 <sup>b</sup>
95	13	>10 <sup>b</sup>	>10 <sup>b</sup>
100	>10 <sup>b</sup>	>10 <sup>b</sup>	>10 <sup>b</sup>

a) Test amounts obtained from ENFSI-DWG Sampling Calculator (version 1.1). Available from: <http://www.enfsi.eu/documents/other-publications> and accessible through SWGDRUG here. (When using the ENFSI-DWG Sampling Calculator, enter the following values: Cell C16 = 5. Other, Cell C9 = 7%, Cell F9 = Particle size, Cell C24 = 2%, Cell C27 = Expected purity)

b) Limited by practical weight determinations.

# SOP-CH-001

## STANDARD OPERATING PROCEDURE

for the

## ANALYSIS OF SUSPECTED CANNABIS PLANT MATERIAL

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## 1.0 Introduction

SOP-CH-001 outlines procedures to distinguish between potential hemp and marijuana as defined in the Agricultural Improvement Act of 2018, H.R. 2, 115th Cong. (2018). A Decision Limit (DL) value for total  $\Delta^9$ -tetrahydrocannabinol (THC) present at 1% is established for field laboratory reporting purposes. (NOTE: Total THC =  $\Delta^9$ -tetrahydrocannabinolic acid (THCA) + THC)

## 2.0 Scope

This procedure:

- Identifies marijuana.
- Applies to plant material units with individual net weights above 100 mg.

NOTE: Units with a net weight below 100 mg may be analyzed using this SOP with supervisory approval.

- Does not apply to seeds, liquids (e.g. oils, creams), edibles, and roots.

## 3.0 Analytical Scheme

- Macroscopic and microscopic examination of plant material
- The 4-Aminophenol (AP) color test
- Gas chromatography-mass spectrometry (GC-MS)

## 4.0 Equipment/Materials/Reagents

- Analytical balance
- Microscope (20x or greater)
- Common glassware and tools:
  - Test tubes (10 x 75 mm, or similar and 13 x 100 mm, or similar)
  - Spot plates (ceramic)
  - Autosampler vials
  - Caps (test tube and autosampler)
  - Volumetric pipettes
  - Cotton plugged pipettes
  - Syringe filters



- Solvents:
  - Ethanol (EtOH) (Reagent grade)
  - Water (H<sub>2</sub>O), deionized
  - Methanol (MeOH) (Analytical grade)
  - Chloroform (CHCl<sub>3</sub>) (Analytical grade)
- 4-Aminophenol (CAS No. 123-30-8)
- 4-Androsten-3,17-dione (CAS No. 63-05-8), internal standard (IS)
- Hydrochloric acid (HCl), concentrated (Reagent grade)
- Sodium Hydroxide (NaOH), pellets (Reagent grade)
- Gas Chromatograph-Mass Spectrometer (see Section 11.1)

## **5.0 Preparation of Reagents/Solutions**

### **5.1 Color Test: 4-AP**

- Reagent A: Dissolve 300 mg 4-aminophenol in 995 mL ethanol and 5 mL 2N HCl. Store in amber glass bottle in refrigerator.
- Reagent B: Dissolve 30 g NaOH in 300 mL water and 700 mL ethanol. Store in amber glass bottle in refrigerator.

### **5.2 GC-MS: Internal Standard Solution (ISS)**

- Dissolve 100 mg 4-androsten-3,17-dione in 1800 mL of MeOH and 200 mL of CHCl<sub>3</sub> (0.05 mg/mL in 9:1 MeOH:CHCl<sub>3</sub>).

## **6.0 Standards and Controls**

### **6.1 Negative Control**

- Color test: Combine an aliquot of reagent A and reagent B in a separate test tube or a separate well in spot plate.
- GC-MS: Inject an aliquot of the ISS as negative control.

### **6.2 Positive Control**

- Color Test: See ADM 1-11.
- GC-MS: Prepare a 0.1 mg/mL solution of THC reference material (RM) using the ISS as the solvent.

- Using Class-A volumetric glassware, prepare a 1-1.5 mg/mL THC Stock A solution by transferring 1 mL from the THC RM (~10 mg/mL) into a tared flask or beaker. Evaporate to dryness and weigh the amount of residue remaining. Dilute using the appropriate volume of ISS. For example, if 9.6 mg of residue remain, dilute using 9 mL of ISS.
- Prepare the working THC positive control solution by performing a 1:10 dilution of the THC Stock A solution using ISS.

NOTE: Class-A glassware may be used for the preparation of the working THC solution, but it is not required.

- Analyze the final solution using the THCSRN\_MS01 method.
- Acceptance criteria: THC:IS ratio >1.
- Ensures the stock and working THC positive control solution are used within five weeks from the date of preparation, or until the response (THC:IS ratio) does not meet the acceptance criteria.

NOTE 1: Store all solutions in the refrigerator.

NOTE 2: Mass spectral positive control data is obtained during method validation.

NOTE 3: Alternatively, if plant material of known THC content is available, prepare a positive control sample following the procedures listed in Section 9.6.2.

## **7.0 Calibration**

- Not applicable

## **8.0 Sampling**

- See ADM 2-4. Do not dry plant material. Sample from buds and leaves of the plant material. Do not sample from seeds, stalks, stems, or roots.

## **9.0 Procedure**

- Open, process, and sample evidence following procedures in ADM.
- Conduct macroscopic/microscopic examination on each unit.

NOTE: Effervescence of the calcium carbonate crystal (i.e., cystolith at the base of the hair) in dilute acid may be performed.

- Perform 4-AP color test on each selected unit in the exhibit:
  - Perform one negative control for each laboratory exhibit. Observe color. If color changes, immediately stop analysis; consult with supervisor for further guidance.

- Place approximately 5 mg of sample from each unit in a separate test tube or spot plate well. Cover the sample with reagent A. Add 2-4 drops of reagent B. Observe color change. Document reagent name and lot number or laboratory traceable number in the Remarks section of *4-AP Color Test* in LIMS.
- Analyze each selected unit by GC-MS:
  - Analyze one positive control (Section 6.2) prior to each sample sequence using method THCSCRN\_MS01.

NOTE: The data from the positive control may be used for all exhibits run during a sequence.
  - Perform one negative control for each laboratory exhibit. Analyze negative control by GC-MS using method THCSCRN\_MS01 immediately prior to sample analysis.
  - Using an appropriate method in the balance software, weigh 40-50 mg of sample from each selected unit in separate test tubes. The bud/leaf sample may be broken-up or crumbled with gloved fingers. Add 5 mL of ISS to test tube. Extract for 10 minutes; vortex/mix for 10-15 seconds at least twice during extraction period. Filter each sample solution through a cotton plugged pipette or syringe filter into new auto sampler vial. Analyze each sample by GC-MS using method THCSCRN\_MS01. Visually inspect peak heights of THC and IS to evaluate DL criterion. Document the results in Remarks of *GC-MS Analysis* test in LIMS (e.g., "3:1 THC:IS ratio, THC ≥1%").
- Process evidence after analysis and reseal following procedures in the ADM.

## 10.0 Calculations

- Not applicable

## 11.0 Instrumental Conditions

### 11.1 Gas Chromatography-Mass Spectrometry (GC-MS):

- Method Name: THCSCRN\_MS01
- Instrument: Agilent 7890/5977 GC-MSD, or equivalent
- Column Type and Dimensions: HP-5ms/DB-5ms, 15 m x 0.25 mm x 0.25 µm (or equivalent)
- Inlet Temperature: 250 °C
- Injection Mode: Split
- Maximum Split Ratio: 50:1
- Minimum Injection Volume: 1 µL
- Carrier Gas and Flow: Helium; 1.2 mL/min (4.3 min hold) ramped to 1.5 mL/min at 2 mL/min per min

- Control Mode: Ramped flow
- Oven Program: 210 °C initial temperature ramped to 235 °C at 30 °C/min; hold at 235 °C for 3 min; ramp to 280 °C at 30 °C/min
- Minimum Run Time: 5.3 min
- Ionization Mode: EI (electron ionization)
- Scan Range: 40 – 500 m/z
- Scan Rate: 1,562 amu/s; N = 2
- MS Source Temperature: 230 °C
- MS Quadrupole Temperature: 150 °C
- Transfer Line Temperature: 280 °C
- Tune Type: Standard Tune

## 12.0 Decision Criteria

### 12.1 Qualitative

- Macroscopic/Microscopic Examination - Positive result includes observation that the gross form of the substance is plant material and microscopic observation of cystolithic hairs.

NOTE: Plant material samples that do not contain cystolithic hairs are not covered by SOP-CH-001.

- 4-AP Color Test - Upon addition of reagent B:
  - Blue color observed: indicates total THC > cannabidiol (CBD)
  - Pink color observed: indicates total THC < CBD
- GC-MS - Follow acceptance criteria for gas chromatography (ADM 2-5.10.4) and mass spectrometry (ADM 2- 5.10.9). Visually inspect/compare peak heights of THC and IS.
  - If THC peak height is greater than IS peak height, then total THC is equal to or greater than the DL. Document the results in Remarks of *GC-MS Analysis* test in LIMS (e.g., "3:1 THC:IS ratio, THC≥1%").
  - If THC peak height is less than IS peak height, then total THC does not exceed the DL. Document the results in Remarks of *GC-MS Analysis* test in LIMS (e.g., "0.5:1 THC:IS ratio, THC<1%").

## **12.2 Quantitative**

- Not applicable

## **13.0 Uncertainty of Measurement**

- Not applicable

## **14.0 Reporting**

Include sampling reporting statements per ADM 2-4 in the Exhibit Analysis section of DEA-113.

### **14.1 Positive Results in All Units Tested**

Report "Marijuana" on DEA-113 when the following results are obtained in all units tested:

- Cystolithic hairs observed
- Blue color test results
- THC  $\geq 1\%$  in GC-MS test

### **14.2 Inconclusive Results in all Units Tested**

Report "Inconclusive" on the DEA-113 when any one of the following results are obtained for one or any of the units tested:

- Pink color test result

OR

- THC  $< 1\%$  in GC-MS test

Add Remark: "Inconclusive marijuana/hemp; pending further analysis upon request" in the Remarks section of DEA-113.

### **14.3 Positive and Inconclusive Results**

Report "Marijuana" and "Inconclusive" if:

- Some of the units tested fulfill the criteria in section 14.1 and some of the units tested fulfill the criteria in section 14.2.

Add Remark: "Inconclusive marijuana/hemp; pending further analysis upon request" in the Remarks section of DEA-113.

### **14.4 Negative Results**

If THC is not present at any level:

- Analyze and report substances identified according to ADM 2-5 and 2-11.
  - Report “No Controlled Substances” when no reportable analytes are identified.

## **15.0 Limitations**

Color test produces false positives for dried oregano, dried sage, cannabinol,  $\Delta^8$ -THC, and cannabicyclol.

All Units Positive (Blue, >1% THC)	
Substance Identified	Marijuana
Sampling Statement	
Non-Exemplar	<ul style="list-style-type: none"> <li>• Marijuana confirmed in 1 unit tested of 1 unit received. (<i>Scenario A</i>)</li> <li>• Marijuana confirmed in [#] units tested of [#] units received. (<i>Scenario A</i>)</li> <li>• Marijuana confirmed in [#] units tested of [#] units received indicating, to at least a 95% level of confidence, that at least 90% of the units in the population contain the substance(s). (<i>Scenario B</i>)</li> </ul>
Exemplar	<ul style="list-style-type: none"> <li>• Marijuana confirmed in [#] units tested of [#] units received. (<i>1-9 units</i>)</li> <li>• Marijuana confirmed in [#] units tested of [#] units received. Based on the reported total seizure unit count, this indicates to at least a 95% level of confidence, that at least 90% of the units in the population contain the substance(s). Total seizure unit count not conducted by laboratory personnel. (<i>10 or more units</i>)</li> </ul>

All Units Inconclusive (Blue, <1% THC; Pink, >1% THC; Pink, <1% THC)	
Substance Identified	Inconclusive
Remark	Inconclusive Marijuana/Hemp; pending further analysis upon request.
Sampling Statement	
Non-Exemplar	<ul style="list-style-type: none"> <li>• 1 unit tested of 1 unit received.</li> <li>• [#] units tested of [#] units received.</li> </ul>
Exemplar	<ul style="list-style-type: none"> <li>• [#] units tested of [#] units received.</li> </ul>

Mixture of Results	
<b>Substances Identified</b>	Marijuana and Inconclusive
<b>Remarks</b>	Inconclusive Marijuana/Hemp; Pending further analysis upon request.
Sampling Statement	
<b>Non-Exemplar</b>	<ul style="list-style-type: none"><li>• [#] units tested of [#] units received. Marijuana confirmed in [#] of the [#] units tested.</li></ul>
<b>Exemplar</b>	<ul style="list-style-type: none"><li>• [#] units tested of [#] units received. Marijuana confirmed in [#] of the [#] units tested.</li></ul>



**REDACTED**

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