

Drug Enforcement Administration

Office of Forensic Sciences



Analysis of Drugs Manual

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Table of Contents

CHAPTER 1 – QUALITY ASSURANCE	3
CHAPTER 2 – EVIDENCE ANALYSIS	105
CHAPTER 3 – FIELD ASSISTANCE	179
CHAPTER 4 – FINGERPRINT REDACTED	193
Appendix 1B – Acronyms and Abbreviations	226
Appendix 1C – Instrument Maintenance Schedule	233
Appendix *1D* – Qualitative **and Quantitative** Method Modifications.....	239
Appendix *1E* – Analytical Supplies and Services	241
Appendix 2A – Random Sampling Procedures	242
Appendix 2B – Reporting Statements	244
Appendix 2C – Composite Formation Procedures	248
Appendix *2D* – Uncertainty of Measurement Estimates	251
Appendix *2E* – Instructions for Completing LIMS Tests	259
REDACTED	282
TABLE 1.....	283
TABLE 2.....	284
REDACTED	286
End of Document	287



CHAPTER 1 – QUALITY ASSURANCE

1.0	Quality Assurance	8
1.1	Scope	8
2.0	Proficiency Testing Program	9
2.1	Preparing Proficiency Samples	9
2.1.1	Preparing Proficiency Samples of Known Composition	9
2.1.2	Preparing Proficiency Samples from Evidence	10
2.2	Inter-Laboratory Proficiency Test Samples	11
2.2.1	Preparing PTP Samples	12
2.2.2	Packaging and Distributing of PTP Samples	12
2.2.3	Analyzing PTP Samples and Reporting Results	13
2.3	**Inter-Laboratory Proficiency Test Samples for Weight and Volume Measurement**	16
2.3.1	Preparing PTP Samples	16
2.3.2	Packaging and Distributing of PTP Samples	16
2.3.3	Analyzing PTP Samples and Reporting Results	17
2.3.4	Documenting the PTP	17
2.4	Internal Proficiency Test Samples	19
2.4.1	Preparing IPTP Samples	19
2.4.2	Packaging and Distributing IPTP Samples	20
2.4.3	Documenting IPTP Results	20
2.4.4	Evaluating IPTP Results	21
2.4.5	Reporting IPTP Results	21
2.5	External Proficiency Test Samples	21
2.5.1	Completing an EPTP	21
2.6	Laboratory Blind Proficiency Test Samples	22
3.0	Validating **and Verifying** Qualitative Methods	23
3.1	Validating Qualitative Separation Methods	25
3.1.1	Selectivity	25
3.1.2	Repeatability (Short-term Precision)	28



3.1.3	Reproducibility (Long-term Precision)	29
3.1.4	Ruggedness Testing	30
3.2	Validating Qualitative Confirmatory Methods.....	31
3.2.1	Accuracy.....	31
3.3	**Verifying Qualitative Separation Methods**	34
3.3.1	Selectivity	35
3.3.2	Repeatability (Short-term Precision)	36
3.3.3	Reproducibility (Long-term Precision)	37
3.4	**Verifying Qualitative Confirmatory Methods**	37
3.4.1	Accuracy.....	37
3.5	Validating *and Verifying* the Cannabis Analysis Separatory Method.....	38
3.5.1	Validation Procedures	38
3.5.2	*Verification* Procedures	39
3.6	<i>Ad hoc</i> Validation of Qualitative Instrumental Methods	40
3.7	Validating Qualitative Non-Instrumental Methods	40
3.7.1	Selectivity	41
3.7.2	Limit of Detection	42
3.7.3	Repeatability (Short-term Precision)	43
3.7.4	Reproducibility (Long-term Precision)	43
3.7.5	Ruggedness Testing	45
3.8	Adding Additional Target Analytes	45
3.8.1	Selectivity	45
3.8.2	Limit of Detection	46
3.9	Verifying Qualitative Non-Instrumental methods	47
4.0	Validating **and Verifying** Quantitative Methods.....	48
4.1	*Validating Quantitative Methods*	48
4.1.1	Selectivity	49
4.1.2	Linearity and Limit of Quantitation	52
4.1.3	Repeatability (Short-Term Precision)	54



4.1.4	Accuracy.....	55
4.1.5	Working Range.....	56
4.1.6	Ruggedness Testing	56
4.2	Adding to Validations	57
4.3	Verifying Quantitative Methods	58
4.3.1	Selectivity	59
4.3.2	Linearity and Limit of Quantitation.....	60
4.3.3	Quality Control Check.....	61
4.3.4	Working Range	62
5.0	Validating Quantitative NMR Methods.....	63
5.1	Validation of qNMR Method	63
5.2	Instrument Tests	63
5.2.1	90° Pulse Width and Spectral Width	63
5.2.2	Quantitative Spectral Region Uniformity.....	64
5.2.3	Linearity and Accuracy.....	65
5.2.4	Repeatability	65
5.3	Analyte Specific Tests.....	66
5.3.1	Accuracy and Solubility.....	66
5.3.2	Analyte Stability	66
6.0	Instrument Verification and Maintenance	68
6.1	Procedures and Acceptance Criteria.....	68
6.2	Templates.....	68
6.3	Instrument Performance Verification Procedures and Acceptance Criteria	68
6.3.1	Balances.....	69
6.3.2	Capillary Electrophoresis System.....	73
6.3.3	Gas Chromatography System.....	74
6.3.4	Gas Chromatography-Mass Spectrometry System	75
6.3.5	Gas Chromatography - Infrared Spectrophotometer	76
6.3.6	Infrared Spectrophotometer	77



6.3.7	Ion Mobility Spectrometer	78
6.3.8	Liquid Chromatography System	78
6.3.9	Liquid Chromatography–Mass Spectrometry System.....	80
6.3.10	Nuclear Magnetic Resonance Spectrometer	81
6.3.11	Polarimeter	83
6.3.12	Portable IR or Raman	83
6.3.13	Raman Spectrophotometer	83
6.3.14	Ultraviolet/Visible Spectrophotometer	84
6.3.15	Portable Mass Spectrometer	84
6.4	Documentation Requirements.....	85
6.5	Instrument Maintenance and Scheduling	86
6.5.1	Special Procedures	86
7.0	Field Laboratory Reference Materials	87
7.1	Stock Reference Materials	87
7.2	Working Reference Materials	88
7.3	Storing Controlled Substances and Listed Chemical Reference Materials	88
7.4	Documentation.....	88
7.4.1	Stock Reference Materials	88
7.4.2	Working Reference Materials	89
8.0	Verifying Field Laboratory Reference Materials	92
8.1	Verifying Qualitative Reference Materials	92
8.2	Verifying Quantitative Reference Materials	93
8.3	Verifying QC Samples.....	94
8.4	Verifying NMR Internal Standards	94
8.5	Documentation.....	95
9.0	Monitoring Storage Conditions	96
9.1	Refrigerators and Freezers	96
10.0	Measurement Traceability	98
10.1	Scope	98



10.2	Net Weight.....	98
10.2.1	Balances.....	98
10.2.2	Reference Standards.....	98
10.3	Purity.....	98
10.3.1	Instrumentation and Equipment.....	98
10.3.2	Reference Materials.....	98
10.4	Measurement Assurance.....	99
10.4.1	Metrological Confirmation.....	99
10.4.2	Statistical Control.....	99
10.4.3	Precision and Bias.....	99
10.5	Supplier Evaluation.....	99
10.5.1	Analytical Supplies and Services.....	99
11.0	Reagent Reliability.....	100
11.1	*Preparing Reagents*.....	100
11.2	Documenting Reagents.....	100
11.2.1	Stock Containers.....	100
11.2.2	Secondary Containers.....	100
11.2.3	Commercial (Purchased) Reagents.....	100
11.3	Labeling Containers.....	100
11.4	(Re)Verifying Reagents.....	101
11.5	Disposing of Reagents.....	102
12.0	Peer Review of Forensic Chemist Examinations.....	103
12.1	General Requirements.....	103
12.2	Conducting Peer Reviews.....	103
12.3	Reviewing the Results of the Peer Review.....	103
12.4	Reporting the Results of the Peer Review.....	104



1.0 Quality Assurance

1.1 Scope

The quality assurance program for drug analysis includes:

- A. *Proficiency testing*
- B. *Method validation and verification*
- C. *Instrument verification and maintenance*
- D. *Reference material (RM) verification*
- E. *Storage condition monitoring*
- F. **Measurement traceability**
- G. **Reagent reliability**
- H. Peer review

1.2 Definitions and Abbreviations

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



2.0 Proficiency Testing Program

- A. The Proficiency Testing Program (PTP) consists of four components:
 - 1. Inter-laboratory proficiency testing (PTP)
 - 2. Internal (intra-laboratory) proficiency testing (IPTP)
 - 3. Blind proficiency testing (BPTP)
 - 4. External proficiency testing (EPTP)
- B. PTP and IPTP samples are prepared by DEA laboratories.
- C. BPTP samples are prepared by SFL1.
- D. EPTP samples are obtained from an ***ISO/IEC 17043 accredited provider.*** (See [Appendix *1E*](#))

2.1 Preparing Proficiency Samples

2.1.1 Preparing Proficiency Samples of Known Composition

The PTP Coordinator or designee:

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



5. Calculations (or spreadsheets)
 6. Evaluation process for sample acceptance or rejection
 7. Preparer's identification and date
- E. Analyzes the bulk proficiency sample, prior to distribution.
- F. Identifies or explains sample components.
- G. Quantitates controlled substance(s) or listed chemical(s).
- H. Reviews analytical results for acceptance.
1. Every component is identified or explained.
 2. The calculated purity is $\pm 5\%$, relative to the expected purity.
- I. Rejects proficiency samples not meeting acceptance criteria.
1. 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:

- A. Reviews selected evidence for acceptance against the following criteria:
1. 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.
 2. The original analysis is less than one year old (if possible).
 3. A composite (formed in accordance with the DEA Evidence Sampling Plan) exists of at least:
 - a. PTP: 14.0 g of material available for distribution
 - b. 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.
- B. Removes selected evidence from the vault, in accordance with the laboratory's documented chain-of-custody procedures (i.e., [LIMS](#)).
- C. Obtains a gross weight, prior to opening.



- D. 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.

- E. Adds the *Other Notes* test in LIMS to document the removal of samples for PTP/IPTP preparation.
- F. Separates the composited material from the original packaging.
- G. Homogenizes the composite using techniques appropriate for the sample type, passes the resulting material through a sieve (60-mesh for PTPs and BTPs 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.
- H. Accepts mixtures that continue to appear homogeneous after processing.
- I. Seals rejected mixtures in the original PSEE and returns the exhibit to the vault for destruction.
 - 1. Document the reason for rejection in the *Other Notes* test in LIMS.
- J. Annotates the DEA-48 (in the Remarks section) with the amount of material removed, proficiency designation, date and the preparer's identification.
- K. Reseals any remaining original evidence material and original packaging into the original container (if possible), and obtains a gross weight.
- L. Annotates any repackaging in the *Other Notes* test.
- M. Attaches the DEA-86 (or equivalent format) to the *Other Notes* test.
- N. Returns the remaining original evidence to the vault for destruction.

2.2 Inter-Laboratory Proficiency Test Samples

The LD or designee:

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



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

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

The SFT Section Chief or designee:

- F. Manages the use of PTP samples received.
- G. 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:

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

2.2.2 Packaging and Distributing of PTP Samples

The PTP coordinator or designee:

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



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

- A. 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.
- B. 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.
- C. 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:



- A. Maintains documentation related to each PTP sample originated and each PTP sample analyzed.
- B. 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:

- A. Reviews the results and identifies any qualitative inconsistencies. A qualitative inconsistency exists if any of the following conditions are met:
 - 1. A laboratory reports a controlled substance or **adulterant** not corroborated by at least one other laboratory.



2. A laboratory does not report a controlled substance identified by at least five other laboratories, including the original analysis.
 3. A laboratory does not report an adulterant estimated to be above 1% by at least five other laboratories, including the original analysis.
 4. 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.
- B. 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:

- A. Calculates purity values from each laboratory ****truncated**** to one decimal place.
- B. 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).

- C. Declares an “outlier” when T_n exceeds 2.13 for $n=8$ sample observations or 2.21 for $n=9$ sample observations (T_{critical} at 95% confidence, two-sided test).
- D. Removes ***a single*** “outlier” from the sample data and recalculates the experimental mean. In the event that ***additional outliers appear***, 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.
- E. Establishes a “target range” for the data set. The target range is

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

where $\tilde{x}$ is 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).



- F. Identifies an inconsistency when a quantitative value is determined to be an outlier and falls outside the target range.
- G. 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:

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

2.3 ****Inter-Laboratory Proficiency Test Samples for Weight and Volume Measurement****

****The SFQAM or designee:**

- A. Selects, prepares, and distributes an inter-laboratory proficiency test (PTP) sample for weight and volume measurement to laboratories and SFT.
- B. Ensures PTP samples are distributed in accordance with the accreditation cycle, i.e. one sample per laboratory per cycle.
- C. Sends an email to the SFT Section Chief or designee the same day the PTP sample is distributed, alerting the training facility to expect an inbound delivery.

The SFT Section Chief or designee:

- D. Designates the samples as proficiency tests for the training staff.

2.3.1 **Preparing PTP Samples**

The SFQAM or designee:

- A. Prepares inert material for PTP samples for weight measurement and establishes stable reference unit values.
- B. Purchases certified density reference materials for use as PTP samples for volume measurement.

2.3.2 **Packaging and Distributing of PTP Samples**

The SFQAM or designee:

- A. Labels the PTP containers and PSEEs with the following information:



1. The program name, PTP-W (for weight measurement) or PTP-V (for volume measurement)
 2. The last two digits of the fiscal year
 3. A statement clearly specifying weight or volume measurement only, no chemical analysis conducted
- B. Distributes the PTP samples.
1. Prepares DEA-12 forms for samples to be forwarded to the receiving laboratories and SFT.
 2. Annotates the DEA-12 with the gross weight of the PSEE.
 3. Prepares instruction memoranda to ensure analysis consistency.
 4. Uses registered mail (return receipt requested), or an approved commercial carrier to transfer the sample.

2.3.3 Analyzing PTP Samples and Reporting Results

The PTP coordinator or designee:

- A. Maintains the PTP roster from which a FC is assigned (on a rotating basis) to analyze the PTP sample.
- B. Ensures that the analysis is completed within 30 days following sample receipt.
- C. Posts an electronic PDF of the completed Laboratory Report (DEA-113) and the Forensic Chemist Worksheet (DEA-86) or Case Details Report (CDR) with associated balance data files to the appropriate fiscal year folder on the shared drive by the tenth business day after analyst completion.

NOTE: The DEA-113 will state *Not Applicable* in the Substance(s) Identified table and “Analyzed for weight (and volume) measurement only” in Exhibit Analysis, under Sampling. Only weight (and volume, if applicable) results are reported.

2.3.4 Documenting the PTP

The LD or designee:

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



Originating Office (PTP of Known Weight/Volume)	Analyzing Laboratory
PTP sample number	PTP sample number
The unique identifier of stock material used	Copy of correspondence from the originating office
The manufacturer and lot # of other components used	A completed DEA-113, original worksheets or electronic analytical record, and data
Documents recording the sample preparation and distribution, including weights and packaging descriptions	Copies of DEA-12s documenting transfers of PTP material
Gross weight of the PTP sample PSEEs, after sealing	Copy of the summary report from the originating office
Copies of DEA-12s documenting transfers of PTP material	Documentation that the analyzing chemist received feedback on the results
Measurement results from each laboratory	Destruction authorization
Copy of summary report	Final disposition
Destruction authorization	
Final disposition	

2.3.4.1 Evaluating Weight and Volume Measurements

SFQ evaluates the Weight and Volume PTP results and identifies any inconsistencies.

The SFQAM or designee:

- Ensures all weight measurements are reported to the same precision.
- Ensures all volume measurements are calculated by density determination, per ADM 2-3.2.3
- Calculates the normalized error (E_n) for each reported net weight or determined density using the following equation, where X_{Lab} and U_{Lab} are the laboratory's value and uncertainty, respectively; and X_{Ref} and U_{Ref} are the known reference value and calculated uncertainty, respectively.



$$E_n = \frac{X_{Lab} - X_{Ref}}{\sqrt{U_{Lab}^2 + U_{Ref}^2}}$$

- D. Declares an inconsistency when a reported net weight or determined density produces an E_n value that falls outside the acceptance range (-1 to 1).
- E. Notifies the submitting laboratory of an inconsistency in the results, as soon as possible. (See LOM 71)

2.3.4.2 Reporting Weight and Volume PTP Results

The SFQAM or designee:

- A. Prepares a summary report of the results using the Weight or Volume PTP Summary spreadsheet on the SFDCC.
- B. Disseminates the summary report to the laboratory system by the fifth business day of the succeeding month following posting of laboratory results.
- C. Includes the results and destruction authorization on the quarterly PTP summary report.**

2.4 Internal Proficiency Test Samples

The LD or designee:

- A. Establishes an Internal Proficiency Testing Program (IPTP) for drug analysis within the laboratory.
- B. Assigns one sample each year to each FC on staff that has not completed another proficiency sample (i.e. PTP, BPTP, EPTP).

NOTE: SFT is exempt from IPTP requirements.

1. Selects samples pending destruction that have been analyzed within a year, if possible.
2. 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.

2.4.1 Preparing IPTP Samples

The PTP coordinator or designee:

- A. Prepares IPTP samples of known composition per 1-2.1.1.



- B. Prepares ITP samples from evidence per 1-2.1.2.

***2.4.2* Packaging and Distributing ITP Samples**

The PTP coordinator or designee:

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

***2.4.3* Documenting ITP Results**

The PTP coordinator:

- A. Maintains documentation related to each ITP sample originated and each ITP sample analyzed.
- B. Ensures each file includes the following:
 - 1. The [CDR](#) or a copy of the front and back of the DEA-86 for the original analysis
 - 2. A copy of the annotated DEA-48, or an authorization memo for SFL1
 - 3. The ITP sample number
 - 4. The gross weight obtained prior to breaking the official seal on the original evidence used for the ITP source material
 - 5. Documentation of the amount of material removed from the original evidence, and the disposition of this material
 - 6. Documentation of any repackaging, and the gross weight of original evidence after resealing
 - 7. Copies of DEA-12s documenting transfers of ITP material
 - 8. The CDR and analytical data from the ITP analysis



9. Documentation that the analyzing chemist received feedback on the results

***2.4.4* Evaluating ITP Results**

The PTP coordinator or designee:

- A. Reviews the results and identifies any analytical inconsistencies. An analytical inconsistency exists if any of the following conditions are met:
 1. A controlled substance is reported in only one of the two analyses.
 2. An adulterant estimated to be above 1% is not reported.
 3. 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.
- B. Reports results to laboratory management.

***2.4.5* Reporting ITP Results**

The LD or designee:

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

***2.5* External Proficiency Test Samples**

The LD or designee:

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

NOTE: SFT is exempt from the EPTP requirements.

- B. Receives feedback from SFQ regarding the EPTP results.

***2.5.1* Completing an EPTP**

The LD or designee:



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

***2.6* Laboratory Blind Proficiency Test Samples**

The SFQAM or designee:

- A. 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 ****and Verifying**** Qualitative Methods

****For any method not covered in this section, contact SFM/SFQ for guidance.****

Qualitative method validation and ****verification**** ensures:

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

A qualitative method includes:

- D. The technique (***e.g. separation, confirmation, color test***)
- E. ****Sample preparation procedures****
- F. ***Operating*** parameters
- G. ****Reagent preparation for non-instrumental methods****

Method validation is required for the following:

- H. ***Implementation*** of a newly developed ****or externally published, but non-validated**** method
- I. After modification of method parameters ****that result in a new method**** per **Appendix *1D***

****Method verification is required for the following:****

- J. ***Transfer of an existing validated method to other instruments (within or between laboratories)**
- K. **Implementation of an externally published and validated method (e.g. ASTM test method)***

The LD or designee:

- L. ****Requests approval for qualitative method development in accordance with LOM 76.****
- M. Ensures methods are fit for their intended purpose and used as validated.
- N. Ensures methods are validated prior to use for the identification of controlled and non-controlled substances ****in casework.****
- O. ****Ensures validated methods are verified on each instrument to which they are transferred, prior to the identification of controlled and non-controlled substances.****



P. Ensures validation documentation is updated when new compounds are encountered.

Q. Ensures *ad hoc* validation procedures are performed per *1-3.6.*

The FC:

- R. *Uses verified* laboratory reference materials *for all method validation or verification procedures.*
- S. *Ensures instrument performance verification is completed per 1-6 prior to method validation or verification.*
1. **For new instrumentation, performance verification and validation can be performed simultaneously.**

T. Validates *or verifies* separation methods per *1-3.1 or 1-3.3.*

1. Methods using soft-ionization mass spectrometry with no fragmentation are validated *or verified* per *1-3.2.1.2.1 or 1-3.4*.

U. Validates *or verifies* confirmation methods per *1-3.2 or 1-3.4.*

V. Validates *or verifies* both the separation and confirmation methods of hyphenated instruments.

NOTE: Within a particular instrument, the same **validated or verified** mass spectrometer (MS) or infrared detector (IRD) method may be interfaced with different separation methods.

W. **Validates or verifies non-instrumental methods per 1-3.7, 1-3.8, or 1-3.9.**

X. Prepares a method validation *or verification* report using the template *on the SFDCC.*

Y. Submits a *validation or verification packet* to the Quality Assurance Specialist (QAS) for review.

1. **A validation or verification packet includes:

- a. A final report
- b. A complete spreadsheet(s)
- c. A full instrument method printout
- d. All instrumental data

Z. Submits selectivity or accuracy data for compounds not previously included in the validation to the QAS for review.**

The QAS or designee:



AA. Reviews the ***validation or verification packet for accuracy and completeness.***

BB. Ensures the documentation is retained.

CC. Submits the reviewed ***packet*** to the Laboratory Quality Assurance Manager (LQAM).

DD. ****For compounds not previously included in the validation:**

1. **Updates the master selectivity or accuracy worksheet in the Qualitative Validation Spreadsheet for laboratory-validated methods.**

OR

2. **Provides data to SFQ to update the master selectivity or accuracy worksheet in the Qualitative Validation Spreadsheet for standardized methods.****

The LQAM or designee:

EE. Reviews ***the validation or verification packet,*** approves the report, and makes ***the packet*** available for use by analysts.

FF. Submits the final reports to the Laboratory Document Control Officer (LDCO).

3.1 Validating Qualitative Separation Methods

The FC:

- A. Defines the scope of the method as general-purpose or limited-purpose.
- B. Validates qualitative separation methods - gas chromatography (GC), liquid chromatography (LC), or capillary electrophoresis (CE) - using the following characteristics:
 1. Selectivity
 2. **Repeatability** (short-term precision)
 3. **Reproducibility** (long-term precision)
 4. ****Ruggedness (for standardized methods)****

3.1.1 Selectivity

3.1.1.1 Validation Procedures

****Applies to methods validated after March 16, 2020.****

The FC:



- A. For general-purpose methods, prepares solution(s) containing:
1. ****Dimethyl sulfone, methamphetamine, phenyltetrahydroimidazothiazole (PTHIT), cocaine, heroin, oxycodone, fentanyl, and trazodone.**
- NOTE 1:** Validation may include additional compounds.
- NOTE 2:** When the detector used precludes detection of dimethyl sulfone, the limitation is documented and an alternative compound may be used with SF approval.**
2. Earliest and latest expected eluting compounds ***if not already included in the required set of compounds.***
 3. ****An internal standard as the fixed compound for relative retention time determination.****
 4. A 0.5% controlled substance marker compound (e.g., fentanyl).
 5. If practical and available, associated alkaloids and derivatization byproducts.
- B. For limited-purpose methods (e.g., cannabinoids, enantiomer, steroids, ***late-eluting compounds,*** etc.), prepares solution(s) containing:
1. The target analyte(s) and any potentially related compounds.
 2. ****An internal standard as the fixed compound for relative retention time determination.****
 3. A 0.5% controlled substance marker compound (e.g., fentanyl), if included in the scope of the method.
- C. Prepares solution(s) at concentrations appropriate for the technique.
- D. ***Ensures the low-level marker is not the earliest or latest-eluting compound for methods with four or more target analytes.***
- E. ****Tests compounds not previously included in the validation.**
1. **For relative retention time determination, the same internal standard must be analyzed either as a separate solution or in the same solution with the newly encountered compound.****
- F. Analyzes one injection of the ***negative control with internal standard*** and the test solutions.
- G. ***Determines*** the following:
1. Retention/migration time and relative retention/migration time for each compound.
 2. ***The minimum acceptable retention time of the method where $t_R = 2t_o$.***
 3. Peak-to-peak signal-to-noise (S/N_{pk-pk}) for each compound.



3.1.1.2 Acceptance Criteria

The FC:

A. Evaluates the data and accepts the method when the following are met:

1. For general-purpose methods *dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone* are *detected and are* visually separated from each other and from the internal standard.

****NOTE:** When the detector used precludes detection of dimethyl sulfone and an alternative compound used, the same criteria must be met.**

2. For limited-purpose methods, the tested compounds are visually separated from the target analyte(s) and internal standard.

B. Evaluates the data for each compound tested using the following criteria:

1. A single peak with a clear, non-splitting apex is observed.
2. *Peaks have minimal fronting/tailing.*
3. The first eluting compound *has an absolute retention time $\geq 2t_o$.*
4. A minimum $S/N_{pk-pk} = 3$ is observed, including the 0.5% low-level marker compound.

C. If acceptable data cannot be achieved for a compound, ****documents the compound as a limitation to the method and accepts the method**** as valid for those compounds that have been documented to meet the acceptance criteria.

1. ****For compounds that produce multiple peaks as a result of breakdown, the data generated may be accepted as presumptive. An alternative technique must be used to confirm the intact molecule.****

3.1.1.3 Reporting Requirements

The FC:

A. Documents the selectivity results *on the selectivity worksheet of the Master Qualitative Validation Spreadsheet – Separation Methods include the following:*

1. The retention/migration time (t_R , t_m), relative retention/migration time, and S/N_{pk-pk} for each *compound* analyzed.
2. The minimum acceptable retention time where $t_R = 2t_o$.
3. Method limitations



- B. ****Completes the selectivity section of the report.****

3.1.2 Repeatability (Short-term Precision)

3.1.2.1 Validation Procedures

The FC:

- A. For general-purpose methods, prepares solution(s) containing, at minimum, three compounds plus an internal standard. ***The earliest-eluting***, one middle-***eluting***, and one late-eluting compound must be ***tested.***
- B. For limited-purpose methods, prepares solution(s) containing the target analyte(s) ***plus an internal standard.***
 1. If a limited-purpose method is expected to have more than four target analytes, prepare solution(s) containing, at minimum, three compounds plus an internal standard. ***The earliest-eluting***, one middle-***eluting***, and one late-eluting compound must be ***tested.***
- C. ****Ensures compounds selected have passed acceptance criteria for selectivity.****
- D. Prepares solution(s) at concentrations appropriate for the technique.
- E. Analyzes the solution(s) 30 times in a single sequence using the method being validated, with a ***negative control with internal standard*** prior to the 30 injections.
- F. Measures the retention/migration time (t_R , t_m) and the relative retention/migration time for each compound tested.

3.1.2.2 Acceptance Criteria

The FC:

- A. Evaluates the data for each compound tested using the following criteria:
 1. 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 ***first*** injection.

OR

 2. The individual relative retention/migration times measured are within 1% of ***the first injection.***
- B. ****Limits the method's use according to the following:****
 1. ***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.**



2. 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.
3. 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 analyzed prior to any subsequent batch of injections.*

3.1.2.3 Reporting Requirements

The FC:

- A. Documents the repeatability results *on the repeatability worksheet of the Master Qualitative Validation Spreadsheet – Separation Methods to include the following:*

 1. The retention/migration time and relative retention/migration time for each compound tested.
 2. Method limitations.

- B. **Completes the repeatability section of the report.**

3.1.3 Reproducibility (Long-term Precision)

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

3.1.3.1 Validation Procedures

The FC:

- A. Analyzes *a single injection of* the *repeatability* solution(s) *once per week for six consecutive weeks* with a *negative control with internal standard* prior to each injection.

**NOTE: Six consecutive weeks of an injection encompasses a five week time frame with the first injection in week one as the baseline.
1. In the event data is not collected for one week during the five week time frame, reproducibility testing may continue provided that at least three injections are evaluated for reproducibility data.**
- B. Measures the retention/migration time and relative retention/migration time for each compound analyzed.
- C. Uses the method along with contemporaneous positive controls (i.e. within 24 hours) once selectivity and repeatability testing is complete, but prior to the completion of the reproducibility testing. *Completes a final report and submits to the QAS* upon completion of reproducibility testing.



3.1.3.2 Acceptance Criteria

The FC:

- A. Evaluates the data for each compound tested ***during each week*** using the following criteria:
 - 1. The individual retention/migration times measured during ***weeks 2-6*** are within 0.1 minutes (for GC and LC) or 0.3 minutes (for LC-MS and CE) of the values measured ***for the initial injection (baseline)***

OR

- 2. The relative retention/migration times measured during ***weeks 2-6*** are within 1% of the values measured ***for the initial injection (baseline)***
- B. ****Stops reproducibility testing after the first instance of the data not meeting the acceptance criteria.****
- C. ****Limits the method's use according to the following:****
 - 1. ***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.**
 - 2. **Conversely, if relative retention/migration times do not meet acceptance criteria for repeatability, but absolute retention/migration times do meet the criteria, then absolute retention/migration times must be used for evaluation of reproducibility.**
 - 3. **The method may be accepted as valid for the maximum number of consecutive weeks, up to one month, that have been documented to meet the acceptance criteria, with the last passing injection limiting the method timeframe. During casework analysis, positive controls must then be analyzed within the limited validated timeframe.***

3.1.3.3 Reporting Requirements

The FC:

- A. Documents the reproducibility results ***on the reproducibility worksheet of the Master Qualitative Validation Spreadsheet – Separation Methods** to include the following:
 - 1. The retention/migration time and relative retention/migration time for each compound tested **each week**
 - 2. **Method limitations**
- B. ****Completes the reproducibility section of the report.****

****3.1.4 Ruggedness Testing****



****Ruggedness testing is only applicable to standardized methods validated at SFL1.**

SFQ:

- A. To ensure method transferability, assigns the verification of the validated method to a minimum of two other laboratories.

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

The assigned laboratory:

- B. Performs the verification of the method per 1-3.3.
- C. Submits the final verification packet to SFQ for review.

SFQ:

- D. In coordination with SFL1, evaluates the data and prepares an appendix to the validation report. ******

3.2 Validating Qualitative Confirmatory Methods

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

The FC:

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

3.2.1 Accuracy

3.2.1.1 Validation Procedures

***The FC:**

- A. For general-purpose methods, tests the following:
 - 1. MS: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.
 - 2. Solid-phase IR/Raman: Cocaine HCl, cocaine base, and methamphetamine HCl.



3. GC-IR: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.
4. NMR: Compounds expected to be identified.

NOTE: Validation may include additional compounds.

- B. For limited-purpose methods, tests the target analyte(s).
- C. Tests compounds not previously included in the validation.*
- D. Prepares solution(s) at concentrations appropriate for the detector, *if applicable.*
- E. Analyzes the test sample(s).
- F. Evaluates the data collected against a verified reference database from SFL1, *a commercial library, published literature spectra, or at least one other ISO/IEC 17025-accredited laboratory.*
- G. **For methods validated at SFL1, structural confirmation of the data is acceptable when no external reference is available.**

3.2.1.2 Acceptance Criteria

The FC:

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

3.2.1.2.1 Mass Spectrometry

- A. The overall sample fragmentation pattern (relative ion abundances, m/z values, and isotopic distributions) corresponds to that of the reference spectrum.
 1. **Ensure the spectra have the same base peak. When a single compound exhibits variations in base peak, multiple spectra may be evaluated, documented, and deemed acceptable (e.g., due to spectral tilting, MSMS relative intensities).**
 2. Relative ion abundance is measured with respect to the most intense signal in the spectrum.
 3. **EI-MS spectra obtained in different carrier gases (i.e. helium and hydrogen) are acceptable for comparison provided acceptance criteria is met. If acceptance criteria is not met, EI-MS spectra obtained in the same carrier gas must be used for comparison.**
 4. 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.



5. Collision-induced dissociation (CID) data and source fragmentation data are not evaluated against each other.
- B. The measured m/z values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum.
 1. 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.
 2. 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.
- C. The molecular ion (or pseudo-molecular ion) must be observed in the sample fragmentation spectrum if it is observed in the reference spectrum.
- D. No prominent unexplainable extraneous ions are observed in the sample spectrum.
- E. 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 ***are not confirmatory but*** may be used as separatory techniques and validated ***or verified*** per 1-3. 2.1.2.1 ***or 1-3.4.***

3.2.1.2.2 Infrared Spectroscopy

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

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

3.2.1.2.3 Raman Spectroscopy

- A. The overall sample spectral pattern (relative peak intensities and Raman shifts) corresponds to that of the reference spectrum.
- B. The observed Raman shifts for prominent and well-defined signals in the sample spectrum are within 4 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.

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

3.2.1.2.4 Nuclear Magnetic Resonance Spectroscopy

- A. The overall sample spectral pattern (multiplicity, relative signal intensity, and chemical shifts) corresponds to that of the reference spectrum acquired using the same solvent.
- B. The measured chemical shifts for all signals in the sample spectrum are within 0.2 ppm (^1H -NMR (with the exception of labile proton signals) and 2 ppm (^{13}C -NMR) of those in the reference spectrum.
1. 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.
 2. For other NMR experiments, acceptance criteria must be established within the laboratory and approved by the LD.
- C. No unexplainable extraneous signals are observed in the sample spectrum.

3.2.1.3 Reporting Requirements

The FC:

- A. Documents the accuracy results ***in the Master Qualitative Validation Spreadsheet – Confirmation Methods.***
- B. Documents the reference data (or library) used for spectral comparisons.
- C. **Documents method limitations.**
- D. ****Completes the report.****

3.3 ****Verifying Qualitative Separation Methods****

****The FC:**

- A. **Verifies qualitative separation methods - gas chromatography (GC), liquid chromatography (LC), or capillary electrophoresis (CE) - using the following characteristics:**
1. **Selectivity (selected compounds only)**
 2. **Repeatability (short-term precision)**
 3. **Reproducibility (long-term precision)**



3.3.1 Selectivity

3.3.1.1 Verification Procedures

The FC:

- A. For general-purpose methods, prepares solution(s) containing:
 - 1. Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.

****NOTE:** When the detector used precludes detection of dimethyl sulfone, the limitation is documented and an alternative compound may be used with SF approval.**
 - a. For methods validated prior to March 16, 2020, document in subsequent verification documentation if the method does not detect any of the required compounds.
 - 2. Earliest and latest expected eluting compounds, if not already included in the required set of compounds.
 - 3. The same internal standard or fixed compound as the original validation.
 - 4. The same 0.5% controlled substance marker compound (e.g., fentanyl) as the original validation.
- B. For limited-purpose methods, prepares solution(s) containing:
 - 1. The target analyte(s) for methods with four or less target analytes.
 - 2. The earliest-eluting, a mid-eluting, and a late-eluting compound for methods with more than four target analytes.
 - 3. The same internal standard or fixed compound as the original validation.
 - 4. The same 0.5% controlled substance marker compound (e.g., fentanyl) as the original validation, if included in the scope of the method.
- C. Prepares solution(s) at concentrations appropriate for the technique.
- D. Ensures the low-level marker is not the earliest or latest-eluting compound for methods with four or more target analytes.
- E. Analyzes one injection of the negative control and the test solution(s).
- F. Determines the following:
 - 1. Retention/migration time and relative retention/migration time for each compound.



2. The minimum acceptable retention time of the method where $t_R = 2t_o$.
3. Peak-to-peak signal-to-noise (S/N_{pk-pk}) for each compound.

3.3.1.2 Acceptance Criteria

The FC:

- A. Evaluates and accepts the data when criteria listed in 3.1.1.2 are met.

NOTE: For methods validated prior to March 16, 2020, compounds eluting $< 2t_o$ 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 on the instrument and is documented to fulfill both the repeatability and reproducibility acceptance criteria.

- B. Accepts the verification when the relative retention/migration time for each compound is $< 15\%$ (relative difference) from the relative retention/migration time listed in the master qualitative validation spreadsheet.

3.3.1.3 Reporting Requirements

The FC:

- A. Documents the selectivity results on the Qualitative Verification Spreadsheet – Separation Methods to include the following:
 1. The retention/migration time (t_R , t_m), relative retention/migration time, and S/N_{pk-pk} for each compound tested.
 2. The minimum acceptable retention time where $t_R = 2t_o$.
 3. The original validation relative retention/migration times.
- B. Completes the selectivity section of the report.

3.3.2 Repeatability (Short-term Precision)

3.3.2.1 Verification Procedures

The FC:

- A. Performs repeatability per 1-3.1.2.1 and 1-3.1.2.2.

NOTE: For methods validated prior to March 16, 2020, an internal standard is not required as part of repeatability for verification if not used in the original validation.

- B. Documents the repeatability results on the Qualitative Verification Spreadsheet – Separation Methods to include the following:



1. The retention/migration time and relative retention/migration time for each compound tested.
 2. Method limitations.
- C. Completes the repeatability section of the report.

3.3.3 Reproducibility (Long-term Precision)

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

3.3.3.1 Verification Procedures

The FC:

- A. Performs reproducibility per 1-3.1.3.1 and 1-3.1.3.2.

NOTE: For methods validated prior to March 16, 2020, an internal standard is not required as part of reproducibility for verification if not used in the original validation.

- B. Documents the reproducibility results on the Qualitative Verification Spreadsheet – Separation Methods to include the following:
1. The retention/migration time and relative retention/migration time for each compound tested
 2. Method limitations
- C. Completes the reproducibility section of the report.**

3.4 **Verifying Qualitative Confirmatory Methods**

**The FC:

- A. Verifies confirmatory methods - mass spectrometry (MS), infrared (IR) spectroscopy, Raman spectroscopy, or nuclear magnetic resonance (NMR) spectroscopy - using the following characteristics:
1. Accuracy (selected compounds only)

3.4.1 Accuracy

3.4.1.1 Verification Procedures

The FC:

- A. For general-purpose methods, tests the following:
1. MS: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.



- a. For methods validated prior to March 16, 2020, document if the method was not validated for any of the required compounds.
2. Solid-phase IR/Raman: Cocaine HCl, cocaine base, and methamphetamine HCl.
3. GC-IR: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.
 - a. For methods validated prior to March 16, 2020, document in subsequent verification documentation if the method was not validated for any of the required compounds.
4. NMR: Compounds expected to be identified.
- B. For limited-purpose methods:
 1. Tests all target analyte(s) for methods with four or less target analytes.
 2. Tests four commonly encountered compounds for methods with more than four target analytes.
- C. Prepares solution(s) at concentrations appropriate for the detector, if applicable.
- D. Analyzes the test sample(s).
- E. Evaluates the data collected against a verified reference database from SFL1, a commercial library, published literature spectra, or at least one other ISO/IEC 17025-accredited laboratory.

3.4.1.2 Acceptance Criteria

The FC:

- A. Evaluates the data per 1-3.2.1.2.
- B. Documents the accuracy results in the Qualitative Verification Spreadsheet – Confirmation Methods.
- C. Documents the reference data (or library) used for spectral comparisons.
- D. Completes the report.**

3.5 Validating *and Verifying* the Cannabis Analysis Separatory Method

3.5.1 Validation Procedures

The FC:

- A. Performs and evaluates selectivity per 1-3.1.1.



- B. Performs and evaluates repeatability per 1-3.1.2.
 - 1. Calculates the THC:IS ratio for each of the injections and documents the RSD of the 30 injections.
- C. Performs and evaluates reproducibility per 1-3.1.3.
 - 1. Calculates the THC:IS ratio for each of the injections and documents the RSD over a ***five*** week period.
- D. Evaluates the response (THC:IS ratio) using THC reference material solutions at different concentrations (linear assessment).
- E. Evaluates the response (THC:IS ratio) using cannabis plant material at different THC concentrations (accuracy assessment).
- F. ****Evaluates verification data as part of ruggedness testing.****

3.5.1.1 Reporting Requirements

The FC:

- A. Documents results using the ***SFL1 method specific report template.***
- B. Documents method limitations.

3.5.2 ***Verification* Procedures**

The FC:

- A. Performs and evaluates selectivity per 1-3.1.1.
- B. Performs and evaluates repeatability per 1-3.1.2.
 - 1. Calculates the THC:IS ratio for each of the injections and documents the RSD of 30 injections.
- C. 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.
 - 1. Accepts accuracy assessment data when:
 - a. The THC:IS for the low concentration sample is <1
 - b. The THC:IS for the high concentration sample is >1.
 - 2. The THC:IS for the middle concentration sample is for informational purposes.



3.5.2.1 Reporting Requirements

The FC:

- A. Documents results using the THSCRN Field Labs **Verification** Report.
- B. Documents method limitations.

3.6 *Ad hoc* Validation of Qualitative Instrumental Methods

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

The FC:

- A. Notifies the SC of the need for an *ad hoc* method for analysis.
- B. Develops a new general-purpose or limited-purpose method.
- C. Submits the method parameters to SFQ through the laboratory management chain-of-command.
- D. Initiates validation of a newly developed separation method per 1-3.1.
- E. Initiates validation of a newly developed confirmation method per 1-3.2.
- F. Obtains SC approval to use the new *ad hoc* method for casework.
- G. Documents approval of the deviation using the *Supervisory Approval* or *Deviation* test.
- H. Analyzes the unknown sample and contemporaneous positive controls (i.e. within 24 hours) using the new method per 2-5.
- I. Completes validation and documentation of the new method within 90 days for separation methods or 30 days for confirmation methods.

The SC or designee:

- J. Approves the use of the newly developed *ad hoc* method for casework.
- K. *****Notifies SFQ of the use of the *ad hoc* method and requests approval for qualitative method development in accordance with LOM 76.*****
- L. Ensures full validation procedures and documentation are completed by the required deadlines.

*****3.7 Validating Qualitative Non-Instrumental Methods*****

The FC:



- A. Defines the analyte(s) to be included in the limited-purpose scope of the method.
- B. Validates non-instrumental methods - color, precipitate, microcrystalline, or immunoassay tests - using the following characteristics:
 - 1. Selectivity, to include matrix effects
 - 2. Limit of Detection (LOD)
 - 3. Repeatability (short-term precision)
 - 4. Reproducibility (long-term precision)
 - 5. Ruggedness
- C. Adds target analytes not included in the original validation according to 1-3.8.

NOTE: Validation of TLC is a combination of separatory and non-instrumental procedures. See SFQ for guidance.

3.7.1 Selectivity

3.7.1.1 Validation Procedure

The FC:

- A. Tests a negative control in triplicate.
- B. Tests, at minimum, 20 compounds in triplicate to include the following:
 - 1. Compounds expected to produce positive results.
 - 2. Compounds expected to produce negative results.
 - 3. All target analyte(s) and related compounds.
 - 4. Other controlled substances, adulterants, and diluents routinely found in exhibits containing the target analyte(s).
 - 5. If practical and available, associated alkaloids and derivatization byproducts.
- C. Tests the following six mixtures, at minimum, in triplicate to assess matrix effects and interferences:
 - 1. Each target analyte with an adulterant at 5%, 50%, and 95%.
 - 2. Each target analyte with a diluent at 5%, 50%, and 95%.



NOTE: If more than one target analyte from a class of compounds produces the same result, it is sufficient to test one target analyte in the above mixtures.

3.7.1.2 Acceptance Criteria

The FC:

- A. Accepts results for each compound or mixture tested when the expected result is obtained.
- B. If acceptable results cannot be achieved for a compound or mixture, documents the limitations.

3.7.1.3 Reporting Requirements

The FC:

- A. Documents the selectivity results on the selectivity worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods to include the following:
 - 1. The result (e.g. precipitate formation)
 - 2. Method limitations (e.g. false negative or false positive results)
- B. Completes the selectivity section of the report.

3.7.2 Limit of Detection

3.7.2.1 Validation Procedures

The FC:

- A. Tests at least five replicates of:
 - 1. A negative control
 - 2. Each target analyte at seven decreasing concentration levels, at minimum
- B. Continues testing at decreasing concentration levels until at least one negative result is obtained for each analyte.

3.7.2.2 Acceptance Criteria

The FC:

- A. Evaluates the results for each compound or mixture tested using the following criteria:
 - 1. The expected positive result is obtained.
 - 2. The first negative result is obtained.



3.7.2.3 Reporting Requirements

The FC:

- A. Documents the LOD for each target analyte as the lowest amount where all five replicates produce a positive result on the LOD worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods.
- B. Completes the LOD section of the report.

3.7.3 Repeatability (Short-term Precision)

3.7.3.1 Validation Procedures

The FC:

- A. Tests a negative control.
- B. Tests 30 replicates of at least two target analytes, if applicable, at an amount approximately equal to twice the established LOD (2xLOD).
- C. Tests 30 replicates of at least two compounds (other than low-level target compounds) expected to produce a negative result.
- D. For commercial (purchased) tests, ensures testing encompasses multiple lot numbers.

3.7.3.2 Acceptance Criteria

The FC:

- A. Evaluates the results for each compound or mixture tested using the following criteria:
 - 1. The expected result is obtained.
 - 2. No more than 20% false negative results are obtained for the target analytes.
 - 3. No false positives are obtained.

3.7.3.3 Reporting Requirements

The FC:

- A. Documents the repeatability results on the repeatability worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods.
- B. Completes the repeatability section of the report.

3.7.4 Reproducibility (Long-term Precision)



3.7.4.1 Validation Procedures

The FC:

- A. Tests a negative control.
- B. Tests the following once per month for four consecutive months:
 - 1. At least two target analytes, if applicable, at an amount approximately equal to twice the established LOD (2xLOD).
 - 2. At least two compounds (other than low-level target compounds) expected to produce a negative result.

NOTE: Four consecutive months encompasses a three month time frame with the first month as the baseline.

- C. In the event data is not collected for one month during the four month time frame, reproducibility testing may continue provided that at least three tests are evaluated for reproducibility data.
- D. Uses the method along with contemporaneous positive controls (i.e. within 24 hours) once selectivity and repeatability testing is complete, but prior to the completion of the reproducibility testing.

NOTE: For tests that require contemporaneous positive and negative controls, reproducibility testing is not required.

3.7.4.2 Acceptance Criteria

The FC:

- A. Evaluates the results for each compound or mixture tested using the following criteria:
 - 1. The expected result is obtained.
 - 2. No false negative results are obtained for the target analytes.
 - 3. No false positives are obtained.
- B. Accepts the method as valid for the maximum number of consecutive months, up to three months, that have been documented to meet the acceptance criteria, with the last passing test limiting the method timeframe. During casework analysis, positive controls must be analyzed within the established validated timeframe.*

3.7.4.3 Reporting Requirements

The FC:



- A. Documents the reproducibility results on the reproducibility worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods.
- B. Completes the reproducibility section of the report.

3.7.5 Ruggedness Testing

SFQ:

- A. To ensure method transferability, assigns the verification of the validated method to a minimum of two other laboratories.

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

The assigned laboratory:

- B. Performs the verification of the method per 1-3.9.
- C. Submits the final verification packet to SFQ for review.

SFQ:

- D. In coordination with SFL1, evaluates the data and prepares an appendix to the validation report.**

3.8 Adding Additional Target Analytes

3.8.1 Selectivity

3.8.1.1 Validation Procedures

The FC:

- A. Tests a negative control in triplicate.
- B. Tests the additional target analyte(s) in triplicate.
- C. If the additional target analyte(s) is from a structural class that was not tested during validation, tests the following six mixtures, at minimum, in triplicate to assess matrix effects and interferences:
 - 1. Each target analyte with an adulterant at 5%, 50%, and 95%.
 - 2. Each target analyte with a diluent at 5%, 50%, and 95%.

NOTE: If multiple target analytes from a class of compounds produce the same result, it is sufficient to test one representative target analyte in the above mixtures.



3.8.1.2 Acceptance Criteria

The FC:

- A. Evaluates the results for each compound or mixture tested using the following criteria:
 - 1. A positive result is obtained.
- B. If acceptable results cannot be achieved for a compound or mixture, documents the limitations.

3.8.1.3 Reporting Requirements

The FC:

- A. Documents the selectivity results on the selectivity worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods to include the following:
 - 1. The result (e.g. precipitate formation)
 - 2. Method limitations (e.g. false negative and false positive result)
- B. Completes the selectivity section of the report.

3.8.2 Limit of Detection

3.8.2.1 Validation Procedures

The FC:

- A. Tests at least five replicates of:
 - 1. A negative control
 - 2. Each target analyte at seven decreasing concentration levels, at minimum.
- B. Continues testing at decreasing concentration levels until at least one negative result is obtained for each analyte.

3.8.2.2 Acceptance Criteria

The FC:

- A. Evaluates the results for each compound or mixture tested using the following criteria:
 - 1. The expected result is obtained.

3.8.2.3 Reporting Requirements



The FC:

- A. Documents the LOD for each target analyte as the lowest amount where all five replicates are positive on the LOD worksheet of the Master Qualitative Validation Spreadsheet – Non-Instrumental Methods.
- B. Completes the LOD section of the report.

3.9 Verifying Qualitative Non-Instrumental methods

- A. Verification of color, precipitate, and microcrystalline test methods is accomplished by testing the reagent reliability per 1-11 or testing both contemporaneous positive and negative controls.
- B. Verification of immunoassay methods is accomplished by testing both contemporaneous positive and negative controls.**



4.0 Validating ****and Verifying**** Quantitative Methods

****Quantitative method validation and verification ensures:**

- A. Objective evidence is maintained demonstrating that the method performs as intended.
- B. Analysts are aware of known method limitations.
- C. Methods are capable of determining the purity of the target analyte included within the validation scope.

A quantitative method includes:

- D. The technique
- E. Sample preparation procedures
- F. Instrument parameters

Method validation is required for the following:

- G. Implementation of a newly developed method
- H. After modification of method parameters that result in a new method per Appendix 1D.

Method verification is required for the following:

- I. Transfer of an existing validated method to other instruments (within or between laboratories)
- J. Implementation of an external published method (e.g. ASTM)

The LD or designee:

- K. Requests approval for quantitative method development in accordance with LOM 76.
- L. Submits a newly developed method to SFQ for validation assignment.
- M. Ensures methods are validated or verified prior to use in casework
- N. Ensures validated methods are verified on each additional instrument to which they are transferred, prior to use in casework.**

4.1 ***Validating Quantitative Methods***

The FC:

- A. ****Uses laboratory reference materials or previously authenticated materials for all method validation procedures.**



- B. Ensures instrument performance verification is completed per 1-6 prior to method validation or verification.
 - 1. For new instrumentation, performance verification and validation can be performed simultaneously.
- C. Includes the following information in the method's scope and description:
 - 1. The controlled substance to be quantitated (i.e. target analyte)
 - 2. The type of samples tested during validation and found amenable for quantitation
 - 3. The instrument name and model**
- D. Validates quantitative separation methods using the following ****performance**** characteristics:
 - 1. Selectivity
 - 2. Linearity ****and Limit of Quantitation (LOQ)****
 - 3. Repeatability
 - 4. Accuracy
 - 5. Ruggedness
- E. ****Prepares a method validation report using the template on the SFDCC.****
- F. Submits a validation packet to SFQ.
 - 1. A validation packet includes:
 - a. A final report
 - b. A complete spreadsheet(s)
 - c. A full instrument method printout
 - d. All instrumental data

****SFQ:**

- G. Reviews the validation packet and approves the report.
- H. Posts the report to the SFDCC.**

***4.1.1* Selectivity**



4.1.1.1 Validation Procedures

****4.1.1.1.1 Separatory Methods****

The FC:

- A. Prepares and analyzes a solution containing only the target analyte and a solution containing only the internal standard (if used).
- B. 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.
- C. Calculates peak resolution and evaluates for any interference ****using the following equation**

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

where t_2 and t_1 are retention times of the target analyte and second component, respectively; and $w_{h(2)}$ and $w_{h(1)}$ are their peak widths at half-height.**

- D. 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:
 1. Inject a combined solution of the tested compound, target analyte, and internal standard (if applicable).
 2. Vary the concentration of the second compound (compared to the target analyte) in order to evaluate potential interferences or interactions

****4.1.1.1.2 Ultraviolet/Visible Methods**

The FC:

- A. **Collects the ultraviolet/visible (UV/Vis) spectrum for the selected solvent.**
- B. **Prepares individual solutions in the selected solvent containing:**
 1. **The target analyte,**
 2. **Selected compounds to be tested to include other controlled substances, diluents, and adulterants routinely found in exhibits containing the target analyte, as well as any available alkaloids or manufacturing byproducts related to the target analyte.**
 3. **Multiple two-component mixtures containing the target analyte and each selected compound. In order to allow the comparison of results, keeps the concentration of the target analyte constant for all tests.**



- C. Collects the UV/Vis spectrum for each of the solutions prepared.**

***4.1.1.2* Acceptance Criteria**

****4.1.1.2.1 Separatory Methods****

The FC:

- A. 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.
- B. Ensures the target analyte and internal standard (if used), are resolved ($R \geq 1.5$) from each compound tested.

****4.1.1.2.2 UV/Vis Methods**

- A. Examines the UV/Vis spectra collected for all solutions analyzed.
- B. Documents the concentration and absorbance of the target analyte at the wavelength selected for quantitation analysis
- C. Documents the concentration and absorbance of other individual compounds and mixtures tested at the wavelength selected for quantitation analysis.
- D. For each compound tested, evaluates the absorbance at the selected wavelength to determine any interference.
- E. Accepts compounds with an absorbance <2% relative to absorbance of the target analyte.

NOTE: If interferences are detected, they are documented as limitations.**

***4.1.1.3* Reporting Requirements**

****4.1.1.3.1 Separatory Methods****

The FC:

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

****4.1.1.3.2 UV/Vis Methods**

The FC:



- A. Documents any method limitations due to spectral interferences, limited solubility, etc.**

4.1.2 Linearity and Limit of Quantitation

The linear range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria. **The LOQ represents an estimate of the analyte concentration or sample amount that can be reliably measured with acceptable precision (repeatability) and accuracy (bias).**

4.1.2.1 Validation Procedures

The FC:

- A. Prepares one high-concentration stock solution of the target analyte using an authenticated (or verified) RM and the appropriate solvent.
- B. Includes the internal standard in the solvent used for preparing the stock and RM solutions, if required by the method.
- C. From the stock solution, prepares at least seven RM solutions at different concentration levels, using volumetric or gravimetric dilutions.
- D. **For separatory methods,** injects all prepared RM solutions a minimum of two times each, in random concentration order.
- E. **For UV/Vis methods, measures the absorbance of all prepared RM solutions five times each, in random concentration order (if possible) at the pre-determined wavelength.**

***4.1.2.2* Data Evaluation**

The FC:

- A. Performs a linear regression analysis using all tested concentrations.
- B. Plots the average instrument response or average instrument response ratio (for methods using an IS), y, as a function of concentration, x.
- C. Does not include the origin (y = 0).
- D. Annotates the resulting slope (m), y-intercept (b), and the correlation coefficient (r), where

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

- E. Prepares a sensitivity plot using the following steps:
 - 1. Determine the average sensitivity (response/amount) for each concentration analyzed.



2. Calculate the overall sensitivity across the concentration range tested by averaging the averages obtained in step 1.
 3. Calculate the sensitivity limits by multiplying the overall sensitivity from step 2 by 95% and 105%.
 4. Plot the average sensitivity per concentration, the overall sensitivity average, the 95% limit, and the 105% limit as a function of concentration (using a logarithmic scale).
- F. ****Ensures that there are at least seven passing concentration data points in both the linear and sensitivity plots.**
1. **Remove any failing data points and regenerate the linear and sensitivity plots.****

****4.1.2.3 Limit of Quantitation**

The FC:

- A. Using the linearity data accepted in 4.1.2.2, determines the standard deviation of y-intercept (σ) and m (slope of the linearity function).
- B. Determines the LOQ using the following equation:**

$$LOQ = \frac{10\sigma}{m}$$

***4.1.2.4* Acceptance Criteria**

The FC:

- A. ****Visually inspects the best-fit-plot, documents, and rejects data due to obvious dilution errors, instrument malfunction, etc. ****
- B. Accepts linearity data for concentrations that fall within the 95 - 105% sensitivity limits.
- C. Ensures the final accepted linear range is determined using at least seven concentrations.

***4.1.2.5* Reporting Requirements**

The FC:

- A. Reports each tested solution concentration, average instrument response or instrument response ratio, and average sensitivity result.
- B. Reports the linear regression plot(s) with the slope (m), y-intercept (b), and the correlation coefficient (r).
- C. Reports the sensitivity plot(s).



- D. Reports the established linear range of the method and the LOQ results.**

4.1.3 Repeatability (Short-Term Precision)

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.

4.1.3.1 Validation Procedures

The FC:

- A. Evaluates repeatability by analyzing at least two solutions, representing the low and high ends of the linear range.
- B. Analyzes each solution five times in the same sequence.
- C. Measures the instrument response or instrument response ratio for each analysis.
- D. Evaluates the repeatability of the method by calculating the relative standard deviation (RSD) of the five analyses at each concentration level tested.
- E. ****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 or instrument response ratio, n is the number of determinations and $\bar{X}$ is the mean of the values obtained.**

***4.1.3.2* Acceptance Criteria**

The FC:

- A. Accepts repeatability data that fulfill the 2%RSD requirement for each concentration level tested.

***4.1.3.3* Reporting Requirements**

The FC:

- A. Reports the calculated RSD for each concentration level.
- B. Documents the established repeatability range which fulfills the acceptance requirements.



***4.1.4* Accuracy**

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

4.1.4.1 Validation Procedures

The FC:

- A. Evaluates the entire linear range during accuracy testing.
- B. Prepares three mixtures containing a known amount of target analyte at low, middle, and high purity concentrations (e.g., 5%, 50%, and 90%).
 1. Prepare the mixtures by combining a known amount of the target analyte with commonly encountered adulterants, diluents, alkaloids, and synthetic byproducts, as applicable within the method's scope.
 2. 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.
 3. 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.
- C. 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.
 1. The following table offers examples 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

- D. Quantitates each of the nine solutions using the specified method, and according to 2-6.



- E. If the scope of the method is applicable to pharmaceutical preparations, in addition to the accuracy mixtures:
1. ***Analyzes authentic tablets/capsules, if available, at concentrations representing routinely encountered levels (e.g. 1%, 5%, and 10%).***
 2. ***If authenticated pharmaceutical preparations are not readily available, analyzes mixtures that mimic the pharmaceutical composition at low purities.***
 3. Ensures all tested formulations (e.g., extended release, different tablet composition, etc.) can be accurately quantitated by the method.
 4. Lists formulations that cannot be accurately quantitated as method limitations.
- F. 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.

***4.1.4.2* Acceptance Criteria**

The FC:

- A. Accepts accuracy data where the experimentally measured purity (expressed in % w/w) is within $\pm 5\%$ (relative) from the known purity.

***4.1.4.3* Reporting Requirements**

The FC:

- A. Documents the sample preparation for ***all solutions tested.***
- B. 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.
- C. Reports the established accuracy range which fulfills the acceptance requirements.

***4.1.5* Working Range**

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

The FC:

- A. ***Determines and documents*** the working range of the method, as described in ***1-4.1.2 through 1-4.1.4.***

****4.1.6 Ruggedness Testing****



****SFQ:**

- A. To ensure method transferability, assigns the verification of the validated method to a minimum of two other laboratories.

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

The assigned laboratory:

- B. Performs the verification of the method per 1-4.3.
- C. Submits the results to SFL1.

SFQ:

- D. In coordination with SFL1, evaluates the data and prepares an appendix to the validation report. **

****4.2 Adding to Validations****

The FC:

- A. Performs additional validation for the following:
 - 1. The appearance of a new component, not included in the original selectivity study associated with the method.
 - a. Minimum Required Performance Characteristics: Selectivity (new component alone and with target analyte and internal standard, if used).
 - 2. Appearance of a new pharmaceutical preparation or matrix (to include new release formulations or different compositions). Notify SFQ of the receipt of new preparations.
 - a. Minimum Required Performance Characteristics: Selectivity (if additional components are present that were not included in the original selectivity testing) and Accuracy.

NOTE: New preparations or matrices may require the completion of a full validation per 1-4.1.1 through 1-4.1.6.

- B. Prepares an appendix to the validation report as applicable.
- C. Provides the appendix and data to SFQ

SFQ:

- D. Updates the master selectivity or accuracy worksheet in the Quantitative Validation Spreadsheet for standardized methods.



- E. Updates validation documentation to list any additional limitations observed.
- F. Posts the appendix to the validation report on the SFDCC.

****4.3 Verifying Quantitative Methods****

****The FC:**

- A. Verifies quantitative methods using the following performance characteristics:
 - 1. Selectivity (Separation methods: critical resolution pairs only; UV/Vis: applicable compounds and mixtures only)
 - 2. Linearity and LOQ
 - 3. Quality Control (QC) Check
- B. Prepares a method verification report using the template on the SFDCC.
- C. Submits a verification packet to the Quality Assurance Specialist (QAS) for review.
 - 1. A verification packet includes:
 - a. A final report
 - b. A complete spreadsheet(s)
 - c. A full instrument method printout
 - d. All instrumental data

The QAS or designee:

- D. Reviews the verification packet for accuracy and completeness.
- E. Ensures the documentation is retained.
- F. Submits the reviewed packet to the Laboratory Quality Assurance Manager (LQAM).

The LQAM or designee:

- G. Reviews the verification packet, approves the report, and makes the packet available for use by analysts.
- H. For initial verifications of newly developed methods, submits the verification packet to SFQ for review as part of ruggedness assessment.
- I. For all other verifications, submits the final reports for posting to the SFDCC.



4.3.1 Selectivity

4.3.1.1 Verification Procedures

4.3.1.1.1 Separatory Methods

The FC:

- A. Prepares and analyzes a solution containing only the target analyte and a solution containing only the internal standard (if used).
- B. Individually analyzes a solution(s) containing the selected critical resolution pair(s):
 1. The target analyte and its critical resolution compound
 2. The internal standard (if used) and its critical resolution compound.
- C. Calculates peak resolution and evaluates for any interference using the following equation

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

where t_2 and t_1 are the retention times of the target analyte and tested component, respectively; and $w_{h(2)}$ and $w_{h(1)}$ are their peak widths at half-height.

4.3.1.1.2 Ultraviolet/Visible Methods

The FC:

- A. Collects the UV/Vis spectrum for the selected solvent.
- B. Prepares individual solutions in the selected solvent containing:
 1. The target analyte,
 2. Selected compounds to be tested to include other controlled substances, diluents, and adulterants routinely found in exhibits containing the target analyte, as well as any available alkaloids or manufacturing byproducts related to the target analyte.
 3. Multiple two-component mixtures containing the target analyte and each selected compound. In order to allow the comparison of results, keeps the concentration of the target analyte constant for all tests.
- C. Collects the UV/Vis spectrum for each of the solutions prepared

4.3.1.2 Acceptance Criteria

4.3.1.1.1 Separatory Methods



The FC:

- A. 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.
- B. Verifies that peaks are visually baseline resolved from the target analyte and internal standard.
- C. Ensures the target analyte and internal standard (if used), are resolved ($R \geq 1.5$) from each compound tested.

4.3.1.1.2 UV/Vis Methods

- A. Examines the UV/Vis spectra collected for all solutions analyzed.
- B. Documents the concentration and absorbance of the target analyte at the wavelength selected for quantitation analysis
- C. Documents the concentration and absorbance of other individual compounds and mixtures tested at the wavelength selected for quantitation analysis.
- D. For each compound tested, evaluates the absorbance at the selected wavelength to determine any interference.
- E. Accepts compounds with an absorbance <2% relative to absorbance of the target analyte.

NOTE: If interferences are detected, they are documented as limitations.

4.3.1.3 Reporting Requirements

4.3.1.3.1 Separatory Methods

The FC:

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

4.3.1.3.2 UV/Vis Methods

The FC:

- A. Documents any additional method limitations due to spectral interferences, limited solubility, etc.

4.3.2 Linearity and Limit of Quantitation



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

4.3.2.1 Verification Procedures

The FC:

- A. Performs linearity and LOQ per 1-4.1.2

4.3.2.2 Reporting Requirements

The FC:

- A. Reports each tested solution concentration, average instrument response or instrument response ratio, and average sensitivity result.
- B. Reports the linear regression plot(s) with the slope (m), y-intercept (b), and the correlation coefficient (r).
- C. Reports the sensitivity plot(s).
- D. Reports the established linear range of the method and the LOQ results.

4.3.3 Quality Control Check

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

4.3.3.1 Verification Procedures

The FC:

- A. Prepares two QC solutions at concentrations representing the low and high ends of the working range.
- B. For separatory methods, quantitates each QC solution three times in the same sequence using the test method. For UV/Vis methods, quantitates each QC solution five times using the test method. Measures the instrument response or instrument response ratio for each analysis.

4.3.3.2 Acceptance Criteria

The FC:

- A. Accepts QC check data that fulfill the 2% RSD requirement.
- B. Accepts QC check data where the experimentally measured average purity is within $\pm 5\%$ (relative) from the known purity.

4.3.3.3 Reporting Requirements



The FC:

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

4.3.4 Working Range

The working range from a method verification is established by the validation working range and the verification linear range and LOQ.

The FC:

- A. Determines and documents the working range of the method.

NOTE: The working range is expected to vary for each instrument.**



5.0 Validating Quantitative NMR Methods

The FC:

- A. 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.
- B. Ensures the following when developing qNMR methods:
 - 1. Analyte signals are clear of interferences.
 - 2. RM and internal standard are soluble in the solvent.
 - 3. 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.
 - 4. 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.
 - 5. All qNMR methods incorporate the use of ***internal standards (IS)***.
 - a. The internal standard in the sample solution is the reference standard.
 - b. Select the ***IS*** based upon availability, solubility, inertness, response characteristics, and absence from exhibits containing the analyte of interest.

5.1 Validation of qNMR Method

The FC:

- A. Validates the qNMR method using instrument and analyte-specific tests.
- B. Performs instrument tests once for a specific instrument and its probe.
 - 1. 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.
- C. Performs analyte-specific tests once for a specific compound using each internal standard/solvent combination to be used.
 - 1. 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:

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

5.2.1.2 Acceptance Criteria

The FC:

- A. Uses pulse widths that are $\leq 90^\circ$ and $\leq 10 \mu\text{s}$.
- B. 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:

- A. Prepares a solution containing a compound with one prominent peak (e.g., dimethyl sulfone in chloroform or “doped” D_2O).
- B. Conducts the qNMR experiment on the prepared solution by adjusting the NMR parameters, as follows:
 - 1. Set the delay (D1) to at least $5 \times T_1$ of the prominent peak.
 - 2. Set the number of transients to 1 or more.
 - 3. 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).
 - 4. Acquire the spectrum.
- C. 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.
- D. Calculates the RSD of these peak heights in the range 0-10 ppm.

5.2.2.2 Acceptance Criteria

The FC:

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



5.2.3 Linearity and Accuracy

The FC:

- A. Performs the linearity study using one common RM.
- B. 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.

- C. Acquires one spectrum for each concentration.
- D. 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.
- E. Plots the results (integral values, y, as a function of concentration, x).
- F. Calculates the correlation coefficient (r). (See *1-4.1.2.2*)
- G. Calculates the purity of the analyte for each concentration.

5.2.3.2 Acceptance Criteria

The FC:

- A. Accepts linearity data with a correlation coefficient greater than 0.998.
- B. Accepts accuracy data where the experimentally measured purity is within $\pm 5\%$ relative to the known RM purity.
- C. 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:

- A. Evaluates repeatability by analyzing at least two concentrations representing the low and high end of the linear range.
- B. Performs a total of five quantitative experiments for each concentration.
- C. Calculates the quantitation values (percent purity) for all peak groups of the analyte.



5.2.4.2 Acceptance Criteria

The FC:

- A. Accepts repeatability data that does not exceed 2% calculated RSD for the following:
 - 1. Quantitative results for the integrals in the individual experiment
 - 2. Quantitative results for the same integral in the spectrum for all experiments for the same sample (e.g., the 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:

- A. Performs qNMR experiments on the target analyte RM.
- B. Integrates peak groups and calculates the purity of the standard at each integral.
- C. 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:

- A. 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.
- B. 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:

- A. Performs a qNMR experiment on the target analyte solution used for accuracy (1-5.3.1) after 2 hours or longer.



- B. Compares quantitative results to the original results in 1-5.3.1.2.
- C. 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:

- A. 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:

- A. Adopts and implements the specific procedures listed below for instruments such as NMR, IR, and Raman.
- B. 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 ***and*** Associate Laboratory Director (ALD):

- C. 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:

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

6.3 Instrument Performance Verification Procedures and Acceptance Criteria

The Instrument Monitor:

- A. Performs instrument performance verification according to the procedures detailed in the sections below.
 - 1. 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 *1D*](#)).



2. 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:

- A. 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.
- B. Ensures that the balance and surrounding area are orderly and free from sources of contamination.

6.3.1.2 Calibration Check

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: NIST-traceable weights
- C. Procedure:

The Balance Monitor:

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



6. Checks the ***sensitivity*** of the balance by using the internal balance adjustment or calibration function.
 7. Checks the repeatability of the balance by performing three measurements on each of two different NIST-traceable weights.
 8. When selecting weights for low-mass and high-mass checks, considers the following:
 - a. Checks should reflect the range of typical sample quantities measured on the balance and should encompass as wide a range as possible/practical.
 - b. 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.
 9. ***Zeros*** the balance ****immediately**** prior to ***the first low-mass*** repeatability check ****and the first high-mass repeatability check.**** Does not ***zero*** the balance prior to each subsequent weighing.
 10. Carefully places the weight on the center of the balance pan, closes balance doors, and waits for the balance reading to stabilize.
 11. 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.
 12. Documents the specific reference weights used (serial number, DEA number, or other form of identification) as well as their corresponding conventional masses and class tolerances.
 13. Checks the accuracy of the balance by evaluating each of the three repeatability measurements.
 14. Maintains calibration check data from balance electronically.
- D. Acceptance Criteria:
1. ***Sensitivity***: Verify that the check is successful.
 2. Repeatability: The RSD for each set of three measurements will be $\leq 0.5\%$.
 3. 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	± 0.000010 g
0.00001 g	± 0.00026 g
0.0001 g	± 0.0004 g
0.001 g	± 0.003 g
0.01 g	± 0.06 g
0.1 g	± 0.3 g

E. If the measurements obtained on a balance do not meet the acceptance criteria,

****The Balance Monitor:****

1. Repeats the reference weight measurements following the procedures in 6.3.1.2.
2. Contacts service provider if problem is not resolved.
3. Places balance out-of-service.
4. Returns balance to service when the measurements obtained meet the acceptance criteria.

6.3.1.3 Calibration

The Balance Monitor:

- A. Ensures the balances are calibrated annually by an ISO/IEC 17025-accredited calibration laboratory. (See [Appendix *1E*](#))
- B. Ensures the calibration certificates include, at minimum, the following:
 1. Balance-specific information (DEA number, manufacturer, model, serial number)
 2. Stamp or logo of the accrediting body
 3. Calibration certificate identification number
 4. Balance capacity
 5. Nominal mass
 6. True mass ("as found" and "as left" values)
 7. Tolerance
 8. Uncertainty



9. Adjustments
10. Environmental conditions
11. Test equipment used
12. Calibration procedures

C. Maintains the calibration reports.

6.3.1.4 Reference Weights

The Balance Monitor:

- A. Utilizes appropriate NIST-traceable weights that are selected based on balance precision and readability. The following are acceptable weight classes based on balance readability:
 1. UltraClass – appropriate for balances with 0.00001 g – 0.000001 g readability.
 2. OIML R 111 Class E1, E2, F1
 - a. Class E1 – appropriate for balances with 0.00001 g – 0.000001 g readability.
 - b. Class E2 – appropriate for balances with 0.0001 g – 0.00001 g readability.
 - c. Class F1 – appropriate for balances with 0.01 g – 0.001 g readability.
 3. ANSI/ASTM E617 Class 1-3
 - a. Class 1 – appropriate for balances with 0.0001 g – 0.00001 g readability.
 - b. Class 2 – appropriate for balances with 0.01 g – 0.001 g readability.
 - c. 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.
- B. Utilizes reference weights with current calibration certificates.
 - C. 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).
 - D. 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.



- E. 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:

- A. Ensures the NIST-traceable weights are calibrated once annually by an ISO/IEC 17025-accredited calibration laboratory. (See [Appendix *1E*](#))
- B. Maintains the calibration certificates.

6.3.2 Capillary Electrophoresis System

6.3.2.1 Electrophoresis

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method (e.g., general-purpose, limited-purpose, or quantitative). ****A general-purpose method must be selected when validated on the instrument.****
- C. Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
 - 1. 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.
 - 2. 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.
 - 3. 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.
 - 4. Quantitative: [Calibrant](#), low QC, and high QC solutions for the method selected.
- D. Procedure: Analyze the performance sample one time using the selected analysis method.
- E. Acceptance Criteria:
 - 1. Tested compounds are visually separated.
 - 2. A single peak with a clear, non-splitting apex is observed for each analyte.
 - 3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.



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

6.3.2.2 Diode Array Detector

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

6.3.3 Gas Chromatography System

6.3.3.1 Chromatography

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method on each column (e.g., general-purpose, limited-purpose, or quantitative). ****A general-purpose method must be selected when validated on the instrument.****
- C. Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
 1. 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.
 2. 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.
 3. 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.
4. Quantitative: Calibrant, low QC, and high QC solutions for the method selected.
- D. Procedure: Analyze the performance sample one time using the selected analysis method.
- E. Acceptance Criteria:
 1. Tested compounds are visually separated.



2. A single peak with a clear, non-splitting apex is observed for each analyte.
3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
4. The first eluting compound is retained with an acceptable retention time $\geq 2t_0$.
 - a. ***If t_R is $< 2t_0$,*** 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 *1D*](#))
5. A minimum $S/N_{pk-pk} = 3$ is observed for each compound tested.
6. 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:

- A. 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

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method on each column (e.g., general-purpose or limited-purpose).
****A general-purpose method must be selected when validated on the instrument.****
- C. Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
 1. 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.
 2. 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.
 3. 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.



- D. Procedure: Analyze the performance sample one time using the selected analysis method.
- E. Acceptance Criteria:
 - 1. Tested compounds are visually separated.
 - 2. A single peak with a clear, non-splitting apex is observed for each analyte.
 - 3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
 - 4. The first eluting compound is retained with an acceptable retention time $\geq 2t_0$.
 - a. ***If t_R is $<2t_0$,*** 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 *1D*](#))
 - 5. A minimum $S/N_{pk-pk} = 3$ is observed for each compound tested.

6.3.4.2 Mass Spectrometer Calibration Check

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

6.3.5 Gas Chromatography - Infrared Spectrophotometer

The FC:

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

6.3.5.1 Gas Chromatography

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method on each column (e.g., general-purpose or limited-purpose).
****A general-purpose method must be selected when validated on the instrument.****
- C. Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)



1. 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.
2. 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.
3. 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.

D. Procedure: Analyze the performance sample one time using the selected analysis method

E. Acceptance Criteria:

1. Tested compounds are visually separated.
2. A single peak with a clear, non-splitting apex is observed for each analyte.
3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
4. The first eluting compound is retained with an acceptable retention time $\geq 2 t_0$.
 - a. ***If t_R is $< 2t_0$,*** 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 *1D*](#))
5. A minimum $S/N_{pk-pk} = 3$ is observed for each compound tested.

6.3.5.2 IR Detector

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: A solution containing a semi-volatile substance with a vapor-phase IR spectrum documented in literature.
- C. 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.
- D. 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

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Polystyrene
- C. Procedure:
 - 1. Collect polystyrene transmittance spectrum (8 scans; 4 cm^{-1} resolution)
 - 2. Report the peak positions measured for the following three bands: 3060, 1601, and 1028 cm^{-1}
- D. Acceptance Criteria: Measured peak positions will be within 4 cm^{-1} of above-referenced values.

6.3.6.2 Reflectance Wavelength and Resolution Check (for ATR)

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

6.3.7 Ion Mobility Spectrometer

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

6.3.8 Liquid Chromatography System

6.3.8.1 Chromatography

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method on each column (e.g., general-purpose, limited-purpose, or quantitative). ****A general-purpose method must be selected when validated on the instrument.****



- C. Performance Sample: Prepared, based on the method, in an appropriate solvent, with internal standard (if used)
1. 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.
 2. 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.
 3. 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.
 4. Quantitative: Calibrant, low QC, and high QC solutions for the method selected.
- D. Procedure: Analyze the performance sample one time using the selected analysis method.
- E. Acceptance Criteria:
1. Tested compounds are visually separated.
 2. A single peak with a clear, non-splitting apex is observed for each analyte.
 3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
 4. The first eluting compound is retained with an acceptable retention time $\geq 2t_0$.
 - a. ***If t_R is $< 2t_0$,*** the t_R of the first eluting compound must be within 0.1 minutes of the t_R obtained during the previous month.
 5. A minimum $S/N_{pk-pk} = 3$ is observed for each compound tested.
- F. 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

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



6.3.9 Liquid Chromatography–Mass Spectrometry System

The FC:

- A. 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

- A. Frequency: Monthly and after substantial maintenance
- B. Method: A commonly used method on each column (e.g., general-purpose or limited-purpose).
****A general-purpose method must be selected when validated on the instrument.****
- C. Performance Sample: Prepared based on the method, in an appropriate solvent, with internal standard (if used)
 - 1. 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.
 - 2. 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.
 - 3. 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.
- D. Procedure: Analyze the performance sample one time using the selected analysis method.
- E. Acceptance Criteria:
 - 1. Tested compounds are visually separated.
 - 2. A single peak with a clear, non-splitting apex is observed for each analyte.
 - 3. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks.
 - 4. The first eluting compound is retained with an acceptable retention time $\geq 2t_o$.
 - a. ***If t_R is $< 2t_o$,** the t_R of the first eluting compound must be within 0.3 minutes of the t_R obtained during the previous month.
 - 5. A minimum $S/N_{pk-pk} = 3$ is observed for each compound tested.

6.3.9.2 Diode Array Detector



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

6.3.9.3 Mass Spectrometer Tune

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Laboratory-customized or manufacturer-recommended compound
- C. Procedure: Tune the mass analyzer following manufacturer's instructions.
- D. Acceptance Criteria: Tune results are within manufacturer's specifications.

6.3.9.4 Mass Spectrometer Calibration

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

6.3.10 Nuclear Magnetic Resonance Spectrometer

*6.3.10.1 High Field (superconducting magnet, typically > 200 MHz)

- A. The instrument is operational for proton acquisition if all three proton tests pass.
- B. The instrument is operational for carbon acquisition if the carbon pulse width test passes.
- C. For nuclei other than ^1H and ^{13}C , (i.e. ^{19}F or ^{15}N), either a sensitivity or a pulse width test must pass manufacturer's specifications within a month of acquiring spectra using these nuclei.
- D. These are the minimum required tests. Additional tests may be performed.

6.3.10.1.1 Proton (^1H) Line Shape

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Manufacturer's recommended sample



- C. Procedure: Measure the non-spinning line shape values using a manufacturer's recommended test
- D. Acceptance Criteria: Peak widths for the chloroform signal are equal to or less than the manufacturer's specifications at 50%, 0.55%, and 0.11% peak heights, respectively.

6.3.10.1.2 Proton (^1H) Pulse Width

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Manufacturer's recommended sample
- C. Procedure: Measure the pulse width using a manufacturer's recommended test
- D. Acceptance Criteria: The 90° pulse width is less than or equal to the manufacturer's specification

6.3.10.1.3 Proton (^1H) Sensitivity

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Manufacturer's recommended sample
- C. Procedure: Measure the signal to noise using a manufacturer's recommended test
- D. Acceptance: The signal to noise is greater than or equal to the manufacturer's specification

6.3.10.1.2 Carbon (^{13}C) Pulse Width

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Manufacturer's recommended sample
- C. Procedure: Measure the pulse width using a manufacturer's recommended test
- D. Acceptance Criteria: 90° pulse width is less than or equal to the manufacturer's specifications

6.3.10.1.3 Carbon (^{13}C) Sensitivity (Not required for instruments using an AutoX probe)

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Manufacturer's recommended sample
- C. Procedure: Measure the signal to noise using a manufacturer's recommended test
- D. Acceptance Criteria: The signal to noise is greater than or equal to the manufacturer's specification

6.3.10.2 Low Field (not a superconducting magnet, typically < 200MHz)



- A. **Frequency:** Prior to use and within 24 hours of the start of sample acquisition.
- B. **Performance Sample:** H₂O in D₂O
- C. **Procedure:** Perform a shimming routine
- D. **Acceptance Criteria:**
 - 1. The peak shape at 50% and 0.55% peak heights must be less than manufacturer's specifications
 - 2. The signal to noise must be greater than 75% of the initial value for that sample.*

6.3.11 Polarimeter

- A. **Frequency:** Every six months and after substantial maintenance
- B. **Performance Sample:** Quartz wave plate filter
- C. **Procedure:** Measure the optical rotation of the quartz wave plate at 589.3 nm (sodium D line).
- D. **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

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

6.3.13 Raman Spectrophotometer

6.3.13.1 Wavelength and Resolution Check (A)

- A. **Frequency:** Monthly and after substantial maintenance
- B. **Performance Sample:** Polystyrene
- C. **Procedure:**
 - 1. Collect spectrum of polystyrene (8 scans; 4 cm⁻¹ resolution).
 - 2. Report the peak positions measured for the following three bands: 3054, 1602, and 1001 cm⁻¹.



- D. Acceptance Criteria: Measured peak positions are within 4 cm^{-1} of above referenced values.

6.3.13.2 Wavelength and Resolution Check (B)

- A. Frequency: Monthly and after substantial maintenance
- B. Performance Sample: Caffeine
- C. Procedure:
1. Collect caffeine spectrum (8 scans; 4 cm^{-1} resolution).
 2. Report the peak positions measured for the following three bands: 2957, 1328, and 555 cm^{-1} .
- D. Acceptance Criteria: Measured peak positions are within 4 cm^{-1} of above-referenced values.

****6.3.14 Ultraviolet/Visible Spectrophotometer****

- A. ****Frequency: Monthly and after substantial maintenance**
- B. **Performance Sample: Low QC and high QC solutions for the method selected.**
- C. **Procedure: Analyze the performance sample one time using the selected analysis method.**
- D. **Acceptance Criteria: QC solutions are within $\pm 5\%$ relative to the known prepared purity of the QC sample.****

****6.3.15 Portable Mass Spectrometer****

6.3.15.1 System Check

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

6.3.15.2 Mass Spectrometer Core Calibration

- A. **Frequency: According to manufacturer's instructions.**
- B. **Performance Sample: N/A**
- C. **Procedure: Calibrate the mass spectrometer core following the manufacturer's instructions.**



- D. **Acceptance Criteria: Calibration results are within the manufacturer's specifications.****

6.4 Documentation Requirements

The LD or designee:

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

The Instrument Monitor:

- C. Maintains records and logbooks for calibration checks, performance verifications, and all other repairs and preventative maintenance.
- D. Includes in the logbook, at a minimum, the following:
 - 1. The identity of the item of equipment and its software and firmware version
 - 2. The manufacturer's name, type identification, and serial number or other unique identification
 - 3. ****Location of instrument manual(s) and software****
 - 4. Evidence of verification that equipment conforms with specified requirements
 - 5. The current location
 - 6. Calibration dates, results of calibrations, adjustments, acceptance criteria, and the due date of the next calibration or the calibration interval
 - 7. Documentation of reference materials, results, acceptance criteria, relevant dates and the period of validity
 - 8. The maintenance plan and maintenance carried out to date, where relevant to the performance of the equipment
 - 9. Details of any damage, malfunction, modification to, or repair of, the equipment
- E. Ensures that the instrument computer is password protected to control access to the system from unauthorized users.

The FC:

- F. 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.



- G. If data does not meet acceptance criteria, makes modifications to the method according to [Appendix *1D*](#) or performs routine maintenance to obtain data that meet the acceptance criteria.
 - 1. Includes in the logbook data that does not meet the acceptance criteria.
 - 2. If method modifications are made, analysts must use the method with the updated parameters for sample analysis.
- H. 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.
 - 1. Investigate the nature and cause of any failure, and make the necessary adjustments and repairs to bring the instrument back to operation.
 - 2. 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.
- I. 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:

- A. Documents maintenance in the instrument logbook.
- B. 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.
- C. Performs maintenance of instruments and equipment according to the schedule in [Appendix 1C](#).

6.5.1 Special Procedures

The LD or designee:

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



7.0 Field Laboratory Reference Materials

The LD:

- A. Designates a primary reference materials (RM) monitor who will administer the program.
- B. Designates alternate RM monitor(s).
- C. 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):

- D. Obtains RMs from SFL1, reputable commercial sources per 1-10.3.2, or another DEA laboratory.
- E. Ensures quantitative RMs are certified reference materials (CRM).
 - 1. SFL1-produced CRMs have traceable purity values of 98% or greater ****and an expanded uncertainty of no greater than 1.25%.****
 - 2. SFL1-produced quantitative RMs with a lower purity are used only with LD authorization on a case-by-case basis.
- F. Treats potentially controlled substance analogue RMs as controlled RMs.
- G. Follows stock and working RM storage procedures for QC samples.
- H. Stores, distributes, and accounts for RMs.
- I. Ensures RMs are verified according to 1-8.
- J. Retains verification results in either electronic or hard copy format.
- K. Transfers RMs to another DEA laboratory in accordance with LOM 74.

7.1 Stock Reference Materials

- A. 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):

- B. Classifies stock RMs as quantitative or qualitative materials.



- C. 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

- A. 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):

- B. Limits controlled working RM to frequently accessed materials.
- C. Makes no more than 1.0 g available to analysts as working RMs.
- D. 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):

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

- A. Maintains records regarding the inventory and transactions of controlled substance RMs in a bound index book or in electronic format to include the following:
 - 1. Name of RM
 - 2. Source of RM
 - 3. Date of receipt



4. Net Weight

- a. 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.

5. RM Number (a lot number or unique identifier for each RM)

6. Transaction reason and date (e.g. transferring to a working vial)

- a. 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.
- b. The RM monitor will record the net weight removed, name of recipient and purpose of transfer in the logbook.
- c. Removal of more than 1 g of RM requires supervisory approval.

7. Final Disposition

- B. Retains records for a minimum of three years after the RM is consumed that contain at least the following:

1. RM source and lot number or unique identifier
2. Name of analyst performing verification
3. Verification date
4. Verification procedure(s) used
5. Authentication, verification and re-verification data

7.4.2 Working Reference Materials

The RM Monitor(s):

- A. Maintains a list of working RMs in a bound logbook or electronic format.
- B. Maintains a sign-out sheet for non-controlled working RM vials.
- C. Maintains sign-out sheets for the controlled working RM vials using the Controlled Working RM Log on the SFDCC.
 1. A separate log must be used for each RM number.



- D. Completes an entry in the log when a controlled working RM vial is replenished.
- E. Monthly, identifies controlled working RMs vials that were not accessed in that time-frame.
- F. For controlled working RM vials that were not accessed, makes the determination to:
 - 1. Remove the controlled working RM vial from the collection.
- OR
- 2. Retain the controlled working RM vial in the collection and record the vial weight using, at a minimum, a 3-place balance.
- G. 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.

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

The FC:

- H. Completes the Controlled Working RM Log at the time the material is accessed and returned.
- I. Uses, at a minimum, a 3-place balance to record vial weights.
- J. Maintains possession of the controlled working RM vial.
 - 1. The controlled working RM vial cannot be transferred to another analyst before returning the vial.
- K. Returns the controlled working RM vial within the same business day of receipt.
- L. 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.

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

The LD or designee:

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



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



8.0 Verifying Field Laboratory Reference Materials

- A. 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).
- B. Verifying RMs will not result in revised purity values. (See 1-8.2)

The FC:

- C. Verifies all RMs used for identification or quantitation in a DEA laboratory report prior to initial use.
- D. Re-verifies RMs every three years.
 - 1. 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:

- A. 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.).
- B. At a minimum, verifies the salt form for cocaine HCl, cocaine base, and methamphetamine HCl.
 - 1. Subsequent verifications of salt form are not required after the initial verification.
- C. Verifies optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., methamphetamine).
 - 1. Subsequent verifications of optical isomeric form are not required after the initial verification.
- D. Analyzes the collected data.
 - 1. Compare the spectrum to authentication, previous verification, or literature data.
 - 2. Identify and explain any unexpected components.
- E. Determines if the RM meets the acceptance criteria.
 - 1. 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.
- F. Submits verification data and associated documents to a RM monitor for review.



- G. Places RMs that meet the acceptance criteria in the RM inventory.
- H. Examines the data for RMs that do not meet the acceptance criteria and chooses the appropriate course of action:
 - 1. 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)
 - 2. Remove the RM from the inventory and send to SFL1 for purification and authentication.
 - 3. Conduct further analysis including quantitation.
 - 4. Transfer the RM to the destruction coordinator.

8.2 Verifying Quantitative Reference Materials

The FC:

- A. 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.
- B. At a minimum, performs a qualitative verification per 1-8.1 and a qNMR for the re-verification of a quantitative reference material.
 - 1. 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.
 - 2. qNMR: Quantitate at least one portion of the RM.
 - a. Inspect the qNMR solution visually to ensure the absence of any insoluble material.
- C. If qNMR is unavailable for use, performs separation area percent purity (SAPP) test using appropriate methods for the compound.
 - 1. SAPP: Analyze at least one portion of the RM on a quantitative separation technique (GC-FID, GC-MS, CE, HPLC, or UPLC).
- D. Analyzes the collected data.
 - 1. qNMR: Calculate the purity from an average of at least three integrals.
 - 2. 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.



- a. 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).
 - b. 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.
- E. Determines if the RM meets the acceptance criteria.
1. 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.
 2. 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.
- F. Submits verification data and associated documents to a RM monitor for review.
- G. Places RMs that meet the acceptance criteria in the RM inventory.
- H. Examines the data for RMs that do not meet the acceptance criteria and chooses the appropriate course of action:
1. Perform additional experiments to establish that earlier results were outliers (e.g., error in sample preparation).
 2. 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)
 3. Remove the RM from the inventory and send to SFL1 for purification and authentication.
 4. Transfer the RM to the destruction coordinator.

8.3 Verifying QC Samples

The FC:

- A. At a minimum, performs a confirmation test using an appropriate technique for the compound.
- B. 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:

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

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

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

8.5 Documentation

The RM Monitor(s):

- A. Creates and maintains files for each drug RM to include at least the following:
 - 1. Completed Reference Material Verification Summary Sheet
 - 2. Authentication data provided by SFL1, commercial source, or other DEA laboratory, if applicable
 - 3. Laboratory-generated verification and re-verification data
- B. 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:

- A. 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.

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

The Monitor:

- E. 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.
- F. Uses a thermometer (appropriate for the required temperature range) ****or electronic temperature monitoring system**** to monitor the refrigerators and freezers, according to the criteria shown below.
 - 1. Refrigerator: $>0^{\circ}\text{C}$ - 10°C (32°F - 50°F)
 - 2. Freezer: $\leq 0^{\circ}\text{C}$ (32°F)
- G. Documents the temperature of each refrigerator or freezer on the Temperature Log form on the SFDCC ****or by using an electronic temperature monitoring system.**** at least once each week
- H. Ensures that each storage device is compliant with the listed environmental conditions.
- I. If a storage device is not compliant with the listed environmental conditions:
 - 1. Posts an "Out of Service" sign on the affected equipment, notifying users not to use the equipment.
 - 2. Evaluates the extent of a problem, and determines the cause and resolves the problem, as soon as possible.
 - 3. Immediately informs a laboratory manager if a storage device is not within the designated parameters



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

The LQAM:

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



10.0 Measurement Traceability

10.1 Scope

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

10.2 Net Weight

10.2.1 Balances

The FC:

- A. 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:

- A. 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:

- A. 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:

- A. Uses **certified reference materials** (CRMs) in analytical processes where uncertainty is estimated, per:
 - 1. 1-7.0
 - 2. 1-8.0
 - 3. See SFL1 RM program SOP
- B. Obtains CRMs from sources determined to be reputable, based on factors including, but not limited to:
 - 1. Demonstrated commitment to good laboratory practices



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

10.4 Measurement Assurance

10.4.1 Metrological Confirmation

The FC:

- A. 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:

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

10.4.3 Precision and Bias

SFQ:

- A. 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:

- A. Ensures analytical supplies and services are in accordance with LOM 75.
- B. Provides records of supplier evaluations and a list of approved suppliers to laboratory staff. (See [Appendix *1E*](#))



11.0 Reagent Reliability

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 *Preparing Reagents*

The FC:

- A. Prepares reagents according to ****the method validation documentation.****

11.2 Documenting Reagents

11.2.1 Stock Containers

The FC:

- A. Completes the Reagent Reliability Verification Form on the SFDCC for each stock (primary) container.

11.2.2 Secondary Containers

The FC:

- A. Completes the Reagent Reliability Verification Form - Secondary Containers on the SFDCC for each secondary container prepared from a verified stock container.

11.2.3 Commercial (Purchased) Reagents

The FC:

- A. Completes the Reagent Reliability Verification Form on the SFDCC once the manufacturer seals are broken on a commercial reagent.

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

11.3 Labeling Containers

The FC:

- A. 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.

***11.4* (Re)Verifying Reagents**

The Reagent Monitor(s) ***or designee***:

- A. Applies these requirements to any reagent in any container used for laboratory analysis.
- B. Verifies reagents ***prior to use in casework.***
- C. Verifies reagents using RMs for all compounds identified using this reagent. Ensures that the results are consistent with the data found in approved reference literature or libraries.
- D. Documents all RMs tested on the Reagent Reliability Verification Form in a reagent logbook.
- E. Re-verifies reagents within three months of the previous successful verification.
- F. Takes the following actions if a reagent does not produce expected results during (re)verification:
 1. Stock Containers:
 - a. Dispose of the stock reagent (See 1-11.8)
 - b. Dispose of secondary reagents traceable to the stock
 - c. Notify laboratory staff by e-mail of actions taken
 - d. Record the disposition date on the Reagent Reliability Verification Form
 2. Secondary Containers:
 - a. Dispose of the secondary reagent (See 1-11.8)



- b. Record the disposition date on the Reagent Reliability Verification Form
- c. Determine if the stock is affected and if so, take action as stated above for stock containers
- d. Notify laboratory staff by e-mail of actions taken

The FC:

- G. *Verifies single-use reagents using contemporaneous positive and negative controls.
- H. Documents results in the case file.*

***11.5* Disposing of Reagents**

The Reagent Monitor(s):

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



12.0 Peer Review of Forensic Chemist Examinations

12.1 General Requirements

- A. 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.

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

12.2 Conducting Peer Reviews

The QAS or designee:

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

The FC:

- C. Accesses the case file(s) in LIMS or retrieves the case file(s) of the specified exhibit(s).
- D. 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.
- E. Provides the completed form to the QAS or designee for review.

12.3 Reviewing the Results of the Peer Review

The QAS or designee:

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

The FC:

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



The SC:

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

12.4 Reporting the Results of the Peer Review

The LQAM or designee:

- A. Compiles and maintains records to document which exhibits underwent peer review, including a summary of the findings and corrections made.
- B. Communicates the summary of findings to the entire chemist staff.
- C. 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)



CHAPTER 2 – EVIDENCE ANALYSIS

1.0 Evidence Analysis.....	110
1.1 Scope	110
2.0 Determining Gross Weight and Opening Evidence.....	111
2.1 Determining Gross Weight.....	111
2.2 Opening Evidence.....	111
2.2.1 Properly Sealed Evidence.....	111
2.2.2 Improperly Sealed Evidence	112
2.3 Describing the Evidence	112
2.4 Handling Interior Packaging	113
2.5 Photographing Evidence	113
2.6 Bulk Evidence	114
2.7 Creating Sub-Exhibits (Splits)	114
3.0 Determining Net Weight and Uncertainty of Measurement Estimates	115
3.1 Observing Minimum Weight Thresholds.....	115
3.2 Calculating Net Weight, Solid Dosage Count, and Volume	115
3.2.1 Powders (mixtures of powders and materials), gummy samples, and plant material 115	
3.2.2 Dosage Units	116
3.2.3 Liquids	117
3.2.4 Internal Body Carry Exhibits.....	117
3.3 Uncertainty of Measurement Estimate Determination	118
3.4 Reporting Net Weight and Uncertainty Estimates	118
3.5 Exceptions	119
3.6 Revising UMEs	119
4.0 The Evidence Sampling Plan.....	120
4.1 Sampling for Qualitative Testing (Identification)	121
4.1.1 Exhibits Containing 1-9 Units	121
4.1.2 Exhibits Containing 10 or More Units	121



4.1.3	Other Exemplar Exhibits (non-Plant Material)	123
4.1.4	Residue Exhibits	123
4.1.5	Arbitrary Sampling	124
4.2	Meeting the Objective of the Sampling Plan	124
4.2.1	Positive Results for All Units	124
4.2.2	Negative Result Observed in One Unit	125
4.2.3	Negative Results Observed in Two or More Units	126
4.2.4	No Analytes Observed for All Units	127
4.3	Sampling to Form Composites	127
5.0	Qualitative Analysis	128
5.1	General Analysis Requirements	128
5.2	Minimum Analysis Requirements	129
5.2.1	Single Unit Analysis	129
5.2.2	Multiple Units: Pre-Composite Analysis	130
5.2.3	Multiple Units: Composite Analysis	130
5.3	Identifying Controlled Substances	131
5.4	Determining Isomers	132
5.5	Identifying Adulterants	132
5.6	Identifying Diluents	133
5.7	Analyzing Residue Exhibits	133
5.8	Identifying Opium	133
5.9	Use of Qualitative Tests and Techniques and Acceptance Criteria	134
5.9.1	Color, Precipitate, and Microcrystalline Tests	134
5.9.2	Immunoassay Tests	135
5.9.3	Thin Layer Chromatography	135
5.9.4	Gas Chromatography	135
5.9.5	Liquid Chromatography	136
5.9.6	Capillary Electrophoresis	136
5.9.7	Infrared Spectroscopy	137



5.9.8	Raman Spectroscopy.....	138
5.9.9	Mass Spectrometry	139
5.9.10	Nuclear Magnetic Resonance Spectroscopy.....	140
6.0	Quantitative Analysis.....	142
6.1	General Requirements.....	142
6.2	Quantitative Procedures.....	143
6.3	Calibrant Solutions.....	143
6.3.1	Separation Methods.....	143
6.3.2	qNMR Methods	144
6.3.3	UV/Vis Methods	144
6.4	Quality Control Samples	145
6.5	Sample Preparation and Analysis	145
6.5.1	Performing a Quantitative Analysis Using a Separation Method	147
6.5.2	Performing a Quantitative Analysis Using qNMR	147
6.5.3	Performing a Quantitative Analysis Using UV/Vis	147
6.6	Acceptance Criteria.....	148
6.7	Reporting Quantitation Results	148
7.0	Determining the Uncertainty of Measurement Estimates for Quantitative Values.....	150
7.1	Reporting UME	150
7.2	Revising UMEs	150
8.0	Processing Evidence After Analysis	151
8.1	Resealing Evidence: Plastic Sealed Evidence Envelopes	151
8.2	Resealing Evidence: Other Evidence Containers	152
8.3	Resealing Bulk Exhibits	153
8.3.1	Submissions by DEA and Other Department of Justice Agencies	153
8.3.2	Submissions by Other Agencies	154
8.4	Creating Additional/New Evidence Containers.....	154
9.0	Reanalyzing Evidence.....	156



9.1	Reanalyzing DEA Exhibits Originally Analyzed in LIMS	156
9.1.1	Reanalysis	156
9.1.2	Reporting	157
9.2	Reanalyzing DEA Exhibits Not Originally Analyzed in LIMS	157
9.2.1	Reanalysis	157
9.2.2	Reporting	158
9.3	Reanalyzing Non-DEA Exhibits	158
9.3.1	Reanalysis	158
9.3.2	Reporting	159
10.0	Removing Sample for Defense Analysis, Reweighing Evidence, and Assessing Evidence Returned from Court	160
10.1	Removing Samples(s) for Defense Analysis from DEA Exhibits Originally Analyzed in LIMS	160
10.1.1	Removing Sample(s)	160
10.1.2	Reporting	162
10.2	Removing Samples(s) for Defense Analysis from DEA Exhibits Not Originally Analyzed in LIMS	162
10.2.1	Removing sample(s)	163
10.2.2	Reporting	164
10.3	Removing Samples(s) for Defense Analysis from Non-DEA Exhibits	165
10.3.1	Removing sample(s)	165
10.3.2	Reporting	166
10.4	Reweighing Evidence	167
10.5	Assessing Evidence Returned from Court	168
	10.5.1 Assessing Evidence When the Outer Seals Were Altered or Broken	168
11.0	The Analytical Record	170
11.1	General Instructions	170
11.2	Completing LIMS Tests	170
11.3	Supporting Data	172
11.4	The Laboratory Report (DEA-113) – General Information	173



11.4.1	The Laboratory Report (SFL1 only).....	175
11.5	The Laboratory Report (DEA-113) – Remarks	175
11.6	Reviewing Analysis and Laboratory Report.....	176
11.7	Analyzing and Reporting Foreign Drug Samples (SFL1 only)	177
11.8	Revising Laboratory Reports (DEA-113)	177



1.0 Evidence Analysis

1.1 Scope

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

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

¹ 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):

- A. ****Ensures all labels on the evidence are consistent with the associated paperwork.****
- B. 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.

- C. 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.
 1. 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.
 2. Take appropriate follow-up action, which may include referral to the Office of Professional Responsibility (OPR).
- D. Reports the gross weight in [LIMS](#) and on the DEA-113.

2.2 Opening Evidence

2.2.1 Properly Sealed Evidence

The FC:

- A. 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.
 1. Annotate FC's initials, date opened, and a [unique identifier](#) on the strip.
 2. Place the annotated strip inside the original evidence envelope.
 3. Annotate the PSEE label with the date opened and any other applicable information.
 4. Record the original condition of the seal and the date opened in the *Description of Evidence* test.
- B. Opens boxes and cans by breaking the SA's, TFO's, or DI's affixed evidence seal(s).



1. Annotate the affixed evidence label with the date opened and any other applicable information.
 2. Record the original condition of the seal(s) and the date opened in the *Description of Evidence* test.
- C. ****Refers to LOM 77 for the decontamination of biohazard exhibits.****

2.2.2 Improperly Sealed Evidence

The FC:

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

The SC:

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

2.3 Describing the Evidence

The FC:

- A. 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).

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

1. 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.

2. Discuss the discrepancy with an SC and contact the SA, TFO, or DI, if necessary, in an attempt to resolve any significant differences.
3. Document the results of the contact in LIMS.



- D. 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:

- A. 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.
1. 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:

- A. Uses a digital camera or digital video camera.
- B. 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).
- C. Photographs evidence during analysis in the laboratory when required, when necessary to document any unusual physical feature(s) prior to processing, or for *REDACTED*.
- D. Attaches all photographs *REDACTED* to the *Image* finding in the *Description of Exhibit and Sampling* test.
- E. Includes a self-documenting sign in all photographs *REDACTED* which contains the following:
1. Unique identifier
 2. Date photographs are taken
 3. Location of the seizure (bulk evidence only)
 4. Laboratory
 5. Handwritten initials of photographer
- F. Positions the sign so that it appears in all the photographs.



- G. Positions an object used to measure the physical size of the seizure, such as a ruler or yardstick, in all photographs.
- H. Assembles or stacks the evidence, when appropriate, so that it makes a clear, visual display of the individual units.
- I. 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:

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

2.7 Creating Sub-Exhibits (Splits)

The FC:

- A. Creates a new [sub-exhibit\(s\)](#) through *Organize My Work, Add Lab Exhibit*.
- B. Enters the *Lab Exhibit* as “*Exhibit Number.0X*” (e.g., 1.01, 1.02, etc.) in the *Lab Exhibit Details* section.
- C. Describes the sub-exhibit's innermost packaging (if applicable) and appearance in the *Description*.
- D. Selects the radial button for *Place in Current Container*.
- E. 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:

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

OR

- B. 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

- C. 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).

- D. 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:

- A. Ensures that established minimum weight thresholds are observed when performing net weight measurements. (See [Appendix *2D*](#))

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:

- A. 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:

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

OR



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

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

OR

C. Determines the total weight by **container** extrapolation as follows:

1. Determine the total gross weight of the units by direct measurement of all units.
2. Individually weigh nine randomly selected empty containers. (See 2-3.5, Policy exception #2)
3. Calculate the average weight per empty container.
4. Obtain the total weight of the empty containers by multiplying the average weight per container by the total number of units in the exhibit.
5. 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:

- A. Counts and weighs all units directly.

3.2.2.2 Ten or more units per exhibit or sub-group

The FC:

- A. Counts and weighs all units directly.

OR

- B. Weighs all units directly; and
- C. Determines the total unit count by extrapolation, as follows:



1. Individually weigh nine randomly selected dosage units.
2. Calculate the average weight per dosage unit.
3. Obtain the total number of dosage units in the exhibit by dividing the total net weight by the average weight per dosage unit.

OR

- D. 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:

- A. Determines the total net weight by direct measurement of the contents of all units.
- B. 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.
- C. 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:

- A. Determines the total net weight by direct measurement of the contents of all units.
- B. 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.
- C. Calculates the total net volume using the total net weight and the composite's density.

OR

- D. Determines the total net weight and volume by **container** extrapolation. (See 2-3.2.1.2)
- E. 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.
- F. Calculates the total net volume using the total net weight and the composite's density.

3.2.4 Internal Body Carry Exhibits

The FC:



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

OR

- B. 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:

- A. 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.

- B. Accepts the calculated uncertainty when:

1. For extrapolation cases, the **relative standard deviation** (RSD) obtained from the nine individual weights measured is 10% or less.
2. The calculated expanded relative uncertainty (U/NW) associated with the total net weight is 25% or less.

- C. 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:

- A. Reports all final net weight and uncertainty results in LIMS and on the DEA-113.

- B. Reports the final **expanded uncertainty** values after rounding to one significant figure using ISO/NIST rounding rules:

1. 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.
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.
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



4. 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).
- C. Reports the final net weight to the same precision as the final expanded uncertainty (same number of decimal places or same level of significance).
- D. 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.

- E. 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

- A. 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.
- B. 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.
- C. Policy exception #3: Since uncertainty is not required for exemplar exhibits, for **exemplar** 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):

- A. 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:

- A. Includes procedures and requirements for FCs to report the identification of analyte(s) in a drug exhibit.
- B. Requires a non-statistical sampling approach for submissions having fewer than 10 units.
- C. Requires a statistical sampling approach for submissions consisting of 10 or more units (non-exemplar). This approach:
 - 1. Requires random sampling.
 - 2. Is based on the probability theory of the hypergeometric distribution.
 - 3. Allows a consistent mathematical foundation for conclusions concerning a high proportion of the exhibit's population.
 - 4. 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.
- D. Includes procedures for sampling residue exhibits.
- E. Includes procedures for arbitrary sampling when no inference will be made on the population.

The ESP for Composite Formation and Quantitative Analysis:

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

The FC:

- G. 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.

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



- I. Documents the reason and obtains approval in advance for any deviations from the ESP, according to [Appendix *2E*](#).

The SC:

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

4.1 Sampling for Qualitative Testing (Identification)

4.1.1 Exhibits Containing 1-9 Units

The FC:

- A. Selects all units.
 1. For single unit exhibits, proceed directly to composite formation according to 2-4.3.
****NOTE:** For homogenous, single-unit liquids, the unit is already considered a representative composite.**
 2. For solid dosage units (except capsules), photograph the front and back of an intact unit prior to analysis.
- B. Analyzes each selected unit as directed in 2-5.
- C. Evaluates results to determine if the objective of the ESP has been met (2-4.2).
- D. 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:

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

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

4.1.2.2.2 Multiple Containers

The FC:

- A. Selects the sample size as follows:
 1. Use [Table 1](#) to determine the number of units to be randomly selected based on the total number of units in the exhibit.
 2. 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.

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

4.1.2.3 Plant Materials

4.1.2.3.1 Exemplar Exhibits



****An exemplar refers to a submitted portion of a larger seizure that may or may not be representative of the entire seizure.****

The FC:

- A. 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)

- B. Analyzes each selected unit as directed in 2-5.
- C. 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:

- A. Uses [Table 1](#) to determine the number of units to be randomly selected.
- B. Segregates or labels (e.g., numbers) each unit selected.
- C. Analyzes each selected unit as directed in 2-5.
- D. 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)

****An exemplar refers to a submitted portion of a larger seizure that may or may not be representative of the entire seizure.****

The FC:

- A. 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.

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

4.1.4 Residue Exhibits

The FC:



- A. 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.

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

4.1.5 Arbitrary Sampling

****Arbitrary sampling procedures apply whenever a portion of the entire submission is analyzed and the remaining portion is not analyzed per customer approval.****

The SC:

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

The FC:

- D. Obtains supervisory approval.
- E. Selects at least one unit and designates it as sub-exhibit X.01.
 - 1. ***Analyzes the selected unit(s) as directed in 2-5.**
 - 2. **Forms a composite as directed in 2-4.3.***
- F. Separates remaining units as sub-exhibit X.02 for no analysis.
 - 1. ****If net weight and unit count are obtained, determines both as directed in 2-3.****
- G. Refers to [Appendix 2B](#) (Scenario G) for sampling statement.
 - 1. ****Places the *No Analysis* statement for X.02 in the *Observations, Results, and Conclusions* section of the DEA-113.****

4.2 Meeting the Objective of the Sampling Plan

4.2.1 Positive Results for All Units



The FC:

- A. Concludes that the objective of the ESP has been met when all selected units have tested positive for the same target analyte(s).
 1. ***At least one consistent*** controlled substance is identified in each of the individual units selected for testing.
 2. ****For exhibits containing 2-9 units, when additional controlled substances are confirmed in some but not all units selected for testing:**
 - a. **Separate the exhibit into sub-exhibits.**

OR

 - b. **Report qualitative result(s) as obtained. Refer to Appendix 2B (Scenario C) for the sampling statement.****
 3. 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.
 4. In the absence of controlled substances, ***at least one*** listed chemical OR non-controlled substance is identified in each of the individual units selected for testing.
 5. Refer to [Appendix 2B](#) (Scenarios A and B) for sampling statements.
- B. Forms a composite according to 2-4.3.

4.2.2 Negative Result Observed in One Unit

The FC:

- A. 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.
- B. Proceeds according to one of the following based on the number of units in the original exhibit:
 1. 2-9: Separate the exhibit into sub-exhibits.
 2. 10-59:
 - a. Analyze all units and separate the exhibit into sub-exhibits.



OR

- b. Perform arbitrary sampling according to 2-4.1.5 and create one or more sub-exhibits.
3. 60 or more:
 - a. Use one of the two options listed for 10-59 units.

OR

- b. Report qualitative result(s) as obtained without testing additional units. Refer to [Appendix 2B](#) (Scenario D) for the sampling statement.
- C. Forms a composite according to 2-4.3.
- D. 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:

- A. 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.

- B. Proceeds according to one of the following based on the number of units in the original exhibit:

1. 3-9: Separate the exhibit into sub-exhibits.
2. 10 or more:
 - a. Analyze all units and separate the exhibit into sub-exhibits.

OR

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

OR

- c. 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.
- C. Forms a composite according to 2-4.3.
- D. 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:

- A. Concludes that the objective of the ESP has been met when no reportable analyte(s) are identified in any of the selected units
- B. 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:

- A. Forms a composite according to [Appendix 2C](#) for all exhibits (or sub-exhibits) except for the following:
 - 1. Exhibits that are not amenable to mixing or grinding
 - 2. Residues
 - 3. Sub-lingual films, blotter paper, patches, etc.
 - 4. Plant materials
 - 5. Substances applied to plant materials
 - 6. No analyte(s) observed for all units (See 2-4.2.4)
- B. Analyzes the composite per 2-5.



5.0 Qualitative Analysis

5.1 General Analysis Requirements

The FC:

- A. Uses validated ***or verified*** qualitative methods during casework analysis. **** (See Appendix 1D for acceptable modifications.) ****
 - 1. ****The use of validated or verified quantitative methods for the qualitative analysis of the target analyte of the quantitation method is acceptable provided a positive control is analyzed concurrently.****
 - B. ****Uses a Standard Operating Procedure (SOP) for analysis, when available.****
 - C. Evaluates the data of the unknown for suitability in meeting acceptance criteria in 2-***5.9*** prior to comparison to the known.
 - D. 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.
 - 1. Any data or observation which is inconsistent with the identification must be fully explained, or the compound cannot be reported.
 - E. Compares the sample results to data from **positive controls** (i.e., DEA laboratory reference materials) analyzed under the following conditions:
 - 1. For color, precipitate, ***and microcrystalline tests***: Within three months. (See 1-11)
 - 2. ****For immunoassay tests: Concurrently. (See 2-5.9.2)****
 - 3. For thin-layer chromatography (TLC): Concurrently. (See ***2-5.9.3***)
 - 4. For gas chromatography (GC), liquid chromatography (LC), and capillary electrophoresis (CE) tests: Within a month using the same method and instrument.
 - a. Positive control data must meet the acceptance criteria found in 1-3.1.1.2.
 - 5. For mass spectrometry (MS), infrared (IR) spectroscopy, Raman spectroscopy, and nuclear magnetic resonance (NMR) spectroscopy: Using the same method and instrument.
 - a. 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 ***verification.***
- F. Includes the following in positive control instrumental data: instrument identifier, reference material unique identifier, and date and method of analysis.



- G. For structural elucidation requests, contacts SFL1.
- H. Bases identifications on established acceptance criteria per 2-***5.9***.
- I. Uses negative controls (blanks) with instrumental and chemical tests to verify that the solvents, **reagents**, and instruments are free of contamination.
- J. Analyzes a negative control immediately prior or concurrently (***non-instrumental tests***) to the sample.

NOTE 1: Blanks are not needed between units when analyzing multi-unit submissions with the same exhibit number (including ***exemplar*** 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-***5.9*** for specific negative control requirements for individual techniques.

5.2 Minimum Analysis Requirements

5.2.1 Single Unit Analysis

The FC:

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

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

- C. 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.

- D. 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.



- E. 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:

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

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

- C. 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.

- D. 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.

- E. 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:

- A. Follows the general requirements in 2-5.1.
- B. Tests at least one sampling. The results may be used for any of the following purposes:
 - 1. Identifying additional controlled substance(s)
 - 2. Identifying non-controlled substances or supplementing the partial identification of non-controlled substances during pre-composite testing



3. Evaluating the sample for **adulterants** at or above 1% level
4. Determining salt form of a target analyte
5. Quantifying a target analyte
6. 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:

- A. Identifies controlled substances present in a sample, provided that a sufficient amount of material exists and all requirements have been met.

1. ****Controlled substances are identified in the pre-composite when possible.**

NOTE: If sufficient data is unable to be obtained during initial pre-composite testing for additional controlled substances, identification of the additional controlled substances may be accomplished through composite sampling.**

2. 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.
3. The general analysis requirements and criteria for identifying controlled substances also apply to the identification of potential controlled substance **analogues**.

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

1. The results from testing at least two samplings are used for identification.
 - a. ***Sampling*** must be from either pre-composite or composite, not from a combination of both.

****NOTE:** When sufficient data is unable to be obtained from pre-composite testing to identify any substance(s) and identification occurs during the analysis of the composite, refer to Appendix 2B (Scenario F) for sampling statement.**

2. 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.



3. **NOTE 2:** For newly encountered substances, potential limitations should be considered when selecting an analytical scheme.
- C. Determines salt form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., cocaine and methamphetamine), unless impractical to do so.
 1. Salt form determination may be performed on the composite.
 2. All salt forms determined are reported.

5.4 Determining Isomers

The FC:

- A. When determining positional, geometric, or diastereomeric isomers, identifies and determines the isomer on each unit, provided sufficient amount of material exists.
 1. If sufficient material does not exist, identifies the compound, including determination of positional, geometric, and diastereomeric designation, in the composite.
 2. Identification and determination of the isomer must be from either pre-composite or composite testing, not from a combination of both.
- B. Determines optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected, unless impractical to do so.
 1. Performs optical isomer determination on the composite.
 2. *Determines optical isomeric form of methamphetamine hydrochloride with a concentration greater than or equal to 80% only when requested.*

5.5 Identifying Adulterants

The FC:

- A. Identifies adulterants, if present at a level of 1% or greater.
- B. Makes identifications based on all of the following minimum criteria:
 1. The result from testing at least one sampling is used for identification.
 2. 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.
 3. Results may be from pre-composite testing, composite testing, or a combination of both.



5.6 Identifying Diluents

The FC:

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

5.7 Analyzing Residue Exhibits

The FC:

- A. Follows the general requirements in 2-5.1.
- B. 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

- C. Makes identifications based on all of the following minimum criteria:
 - 1. The result from testing one sampling (e.g., rinse, swab) using a **procedural blank**.
 - 2. 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.
- D. Segregates and retains the procedural blank and sample vials as reserve evidence when there is insufficient material for two samplings.

5.8 Identifying Opium

The FC:

- A. Identifies opium based on all of the following minimum criteria:
 - 1. A macroscopical examination to observe that the gross form of the substance is a gum-like or resinous, brown material.
 - 2. The results from testing at least two samplings are used for identification.

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



3. 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.

4. At least four of the following are confirmed: codeine, morphine, thebaine, papaverine, or noscapine.

B. Reports “Opium” on the DEA-113.

1. Also reports any adulterants identified per 2-5.5.

***5.9* 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:

- A. Uses the qualitative tests and techniques described in this section to identify controlled and non-controlled substances.
- B. Bases identifications on the general acceptance criteria for each test.
- C. Uses spectral processing tools, as needed.
 1. Scale normalization may be applied for comparison of sample spectra and positive controls.
 2. Background subtraction may be applied for comparison of sample spectra and positive controls.
 3. Spectral subtraction may be used to eliminate the influence of interfering or co-eluting substances.
- D. Uses laboratory-specific protocols approved per 1-6 (performance verification) for any qualitative test not included in this section.

5.9.1 Color, Precipitate, and Microcrystalline Tests

The FC:

- A. Uses ***color, precipitate, and microcrystalline tests*** as presumptive techniques in the qualitative analysis of controlled and non-controlled substances.
- B. Accepts a result when the color change, ****precipitate, or crystal**** observed for the sample is consistent with the positive control.



****5.9.2 Immunoassay Tests****

The FC:

- A. ****Uses immunoassay tests as a presumptive technique in the qualitative analysis of controlled and non-controlled substances.**
- B. **Analyzes both a concurrent positive and negative control using the same lot number of immunoassay test as the sample(s).**
 - 1. **A daily positive control solution must be associated with each batch of samples. Multiple batches can use the same daily positive control solution. Negative control, positive control, and sample solutions must be prepared using the same solvent.**
- C. **Accepts a result when the result observed for the sample is consistent with the positive control.****

***5.9.3* Thin Layer Chromatography**

The FC:

- A. Uses TLC as a separation technique in the qualitative analysis of mixtures.
- B. 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.
- C. Accepts a result when:
 - 1. The retention factor of the analyte is within 5% of the positive control.
 - 2. The spot color of the analyte is consistent with the positive control.

***5.9.4* Gas Chromatography**

The FC:

- A. 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.

- B. 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.

- 1. 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.

- C. Accepts a result when a single peak with a clear, non-splitting apex is observed.
- D. ****Accepts a result when peaks have acceptable shape with minimal peak fronting/tailing.****
- E. Accepts a result when the peak-to-peak signal-to-noise (S/N_{pk-pk}) is greater than 3.
- F. 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%.
- G. Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

***5.9.5* Liquid Chromatography**

The FC:

- A. 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.

- B. 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.

- 1. 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.
- C. Accepts a result when a single peak with a clear, non-splitting apex is observed.
- D. ****Accepts a result when peaks have acceptable shape with minimal peak fronting/tailing.****
- E. Accepts a result when the S/N_{pk-pk} is greater than 3.
- F. 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.
- G. Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

***5.9.6* Capillary Electrophoresis**



The FC:

- A. 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.

- B. 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.

1. 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.

- C. Accepts a result when a single peak with a clear, non-splitting apex is observed.

- D. ****Accepts a result when peaks have acceptable shape with minimal peak fronting/tailing.****

- E. Accepts a result when the S/N_{pk-pk} is greater than 3.

- F. 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%.

- G. Accepts a result for co-analysis when the number or area of peaks reflects the known addition.

***5.9.7* Infrared Spectroscopy**

The FC:

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

- B. 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.

- C. 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.

- D. Displays all spectra in the same units (i.e., transmittance, absorbance, or reflectance).
- E. Evaluates the data using the following acceptance criteria:
 - 1. The overall sample spectral pattern (relative peak intensities and wavenumbers) corresponds to that of the positive control spectrum.
 - 2. The observed wavenumbers for prominent, well-defined signals between 2000 cm^{-1} and 650 cm^{-1} in the sample spectrum are within 4 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.
 - 3. The sample spectral pattern between 4000 cm^{-1} and 2000 cm^{-1} corresponds to that of the positive control spectrum.
 - 4. No unexplainable extraneous signals are observed in the sample spectrum.
- F. Accepts results as confirmatory when the above criteria are met. Otherwise, results may be considered presumptive, provided the wavenumber acceptance criterion has been fulfilled.

***5.9.8* Raman Spectroscopy**

The FC:

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

- C. Evaluates the data using the following acceptance criteria:
 - 1. The overall sample spectral pattern (relative peak intensities and Raman shifts) corresponds to that of the positive control spectrum.



2. The observed Raman shifts for prominent and well-defined signals in the sample spectrum are within 4 cm^{-1} 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.

3. No unexplainable extraneous signals are observed in the sample spectrum.
- D. 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.

***5.9.9* Mass Spectrometry**

The FC:

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

***5.9.9.1* MS as Confirmatory**

The FC:

- A. 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:
 1. The overall sample fragmentation pattern (relative ion abundances, isotopic distributions, and m/z values) corresponds to that of the positive control spectrum.
 - a. ****Ensure the spectra have the same base peak unless variations in abundance have been previously documented and met acceptance criteria in 1-3.2.1.2.1 (e.g., due to spectral tilting, MSMS relative intensities).****
 - b. Relative ion abundance is measured with respect to the most intense signal in the spectrum.
 2. 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.
 3. 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.
 4. 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.
 5. The molecular ion must be observed in the sample spectrum if it is expected and observed in the positive control spectrum.



- a. ****Certain chemical compounds may not consistently produce an observable molecular ion (e.g. concentration, fragmentation conditions). In such cases, comparisons can still be conducted based on the fragment ions present in the spectra.****

6. No unexplainable extraneous ions are observed in the sample spectrum.

- B. 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.

***5.9.9.2* MS as Separation**

The FC:

- A. 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:
1. 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.
 2. 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.
- B. Accepts results as presumptive when the above criteria are met.

***5.9.10* Nuclear Magnetic Resonance Spectroscopy**

The FC:

- A. Uses NMR as a confirmatory technique in the qualitative analysis of controlled and non-controlled substances.
- B. 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).
- C. Evaluates the data using the following acceptance criteria:
1. The overall sample spectral pattern (multiplicity, relative signal intensity, and chemical shifts) corresponds to that of the positive control spectrum.
 2. The measured chemical shifts for all signals in the sample spectrum are within 0.2 ppm (^1H -NMR) (with the exception of labile proton signals) and 2 ppm (^{13}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.



3. No unexplainable extraneous signals are observed in the sample spectrum.
- D. 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

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The FC:

- A. Quantitates exhibits that were purchased as a part of an on-going investigation.
 - B. Quantitates exhibits which require the result for statutory considerations (i.e., methamphetamine, amphetamine, oxycodone, PCP, and hydrocodone).
 - C. Does not routinely quantitate samples when the primary controlled substance is estimated to be present below the 1% level.
 - D. Performs quantitative analyses using validated methods, as posted on the Office of Forensic Sciences Document Control Center (SFDCC). (See [Appendix *1D*](#) for acceptable modifications).
 - 1. 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.
 - 2. When a standardized method is not available for the selected technique, a laboratory-validated method must be used.
 - 3. Laboratory-validated methods are transferrable between instruments and laboratories, as long as the ***verification*** requirements (as specified in ***1-4.3***) are fulfilled.
 - E. 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.
 - 1. 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.
 - F. 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.
- G. Performs quantitative analysis of non-controlled substances, including listed chemicals at the discretion of the LD, or when necessary to support investigations.
 - H. Bases all reported purities on reviewable data and observations.
 - 1. Any data or observation which does not correlate with the quantitation results must be fully explained, or the quantitative value cannot be reported.



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

6.2 Quantitative Procedures

The FC:

- A. ****Uses only calibrated equipment for volume measurements associated with calibrant solution preparations, including in the preparation of qNMR internal standard solutions (ISS).****
- B. Uses only Class-A volumetric glassware or calibrated automatic pipettes for volume measurements associated with QC and sample solution preparations.
- C. Applies minimum net weight requirements (2-3.1) to preparation of all quantitation solutions ****including in the preparation of qNMR ISS.****
- D. Equilibrates solutions maintained under refrigeration to room temperature prior to use.
- E. Prepares negative controls (blanks) from the same solvent or internal standard solution used to prepare the quantitative sample.
 1. 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:

- A. Prepares a ***calibrant*** solution using a certified reference material (CRM).
- B. Factors the documented purity of the CRM into the final concentrations of the calibrant solutions.
- C. Ensures that calibrant solutions ****for methods that use an internal standard**** are used for only one month from the date of preparation.
- D. ****Ensures that calibrant solutions for methods that do not use an internal standard are either:**
 1. **Used for only one month from the date of preparation**
 - OR**
 2. **Are checked once a month to demonstrate that the response of the target analyte remains within 5% relative to the initial response measured after preparation.**
 - a. **The checks must be conducted on the same instrument each month, the results of the checks must be documented in a logbook, and supporting data must be maintained.****



- E. 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

- A. Weighs and directly adds to the sample an IS with traceable purity.
1. Factors the documented purity of the IS into the final amount of the IS.

OR

- B. Prepares an ISS using an IS with traceable purity.
1. Factor the documented purity of the IS into the final concentration of the ISS.
- C. Ensures that the ISS is ***either:**
1. **Used for only one month from the date of preparation***
- **OR**
2. **Is checked once a month to verify the continued accuracy of the prepared concentration.**
 - a. The check is conducted by preparing a quantitation solution of a CRM with a known purity value and the ISS being checked. The ISS may continue to be used when the measured purity of the CRM is within 5% relative to the SFL1 or manufacturer reported purity. The results of the checks must be documented in a logbook and supporting data must be maintained.**

****6.3.3 UV/Vis Methods****

****The FC:**

- A. Prepares three calibrant solutions using a certified reference material (CRM).
- B. Factors the documented purity of the CRM into the final concentrations of the calibrant solutions.
- C. Ensures that calibrant solutions are either:
1. **Used for only one month from the date of preparation**

OR



2. Are checked once a month to demonstrate that the response of the target analyte remains within 5% relative to the initial response measured after preparation.
 - a. The checks must be conducted on the same instrument each month, the results of the checks must be documented in a logbook, and supporting data must be maintained.
- D. Establishes a multi-point calibration curve by analyzing each calibrant solution five times, representing the original validated/verified linear range.

NOTE: The linearity function established during the original validation/verification may be used as the multi-point calibration curve (unacceptable data points not included). **

6.4 Quality Control Samples

The FC:

- A. Uses a quality control (QC) sample from SFL1, when available, to prepare quality control (QC) solutions for use as positive controls during quantitative analysis.
 1. 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).
 2. 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.

- B. Prepares two solutions at target analyte concentrations that represent the lower and higher ends of the method's working range.
 1. **For separation techniques, use the same batch (i.e. same specific container) of ISS used to prepare the calibrant solution.**
- C. For NMR quantitation, prepares one solution within the solubility limits of the method.
- D. Uses QC solutions until they are depleted or the results fall outside of the acceptance criteria. (See 2-6.6)
- E. Documents QC solution preparation procedures in the *Quantitation* test or attachments.

6.5 Sample Preparation and Analysis

The FC:

- A. Prepares a solution(s) so that the target analyte concentration is bracketed between the high and low QC solutions concentrations.



1. ****For separation techniques, use the same batch of ISS (i.e. same specific container) used to prepare the calibrant solution.****
- B. ****For NMR quantitation, prepares a solution(s) within the solubility limits of the method.****
- C. For powders, crystalline materials, and solid dosage forms, obtains the test sample amount from the composite as specified below.
 1. 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 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 Forms	
Total Net Weight < 100 mg	Purity testing is not normally performed unless requested by the customer
Total Net Weight ≥ 100 mg	Refer to Table 2

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

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

Liquids/Solutions	
Total Volume < 5 mL	Purity testing is not normally performed unless requested by the customer
Total Volume ≥ 5 mL	Minimum 1.0 mL

- E. 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.
 1. Use the Scenario J purity statement on the DEA-113 (refer to [Appendix 2B](#)).



Gummy	
<5.0 g per Unit	Purity testing is not normally performed unless requested by the customer
≥ 5.0 g per Unit	≥ 0.5 g (sample portions from various randomly selected units)
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:

- A. 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.
 1. 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:

- A. Quantitates an unknown sample using a minimum of a blank, a sample, and a single QC solution.
 1. 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.

****6.5.3 Performing a Quantitative Analysis Using UV/Vis****

The FC:

- A. Quantitates an unknown sample using a valid multi-point calibration curve, a negative control, and a set of bracketing high and low concentration QC solutions.



1. Each solution is analyzed five times.
2. The QC solutions must bracket the unknown sample in concentration (high and low) and in analysis time (before and after).

6.6 Acceptance Criteria

The FC ensures:

- A. Blanks are free of carry-over and contamination.
- B. QC solutions are within $\pm 5\%$ relative to the known prepared purity of the QC sample.
- C. 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%.
- D. ****For UV/Vis, the RSD of the response (Abs) for the five replicate readings of the unknown sample is less than 2%.****
- E. qNMR results are derived from an average of at least three acceptable integrals.
 1. The use of fewer than three integrals must be supported by the peak height quantitation information and approved by the supervisor.
- F. The maximum acceptable quantitative result is ***106%.***

6.7 Reporting Quantitation Results

The FC:

- A. Reports the final purity value as the average of the injections analyzed, provided that the RSD criteria is fulfilled (2-6.6).
- B. Reports the final purity value for NMR as the average of multiple runs (multiple integrals) of the sample preparation.
- C. ****Reports the final purity value for UV/Vis as the average of the five replicates, provided that the RSD criterion is fulfilled (2-6.6).****
- D. Obtains a final purity value as follows if two or more sample preparations or instrumental techniques are used:
 1. Average the purity results obtained for each individual preparation/technique and document each individual average in the [Case Details Report](#) (CDR) or attachments.
 2. Combine averaged purity results only if each individual averaged result falls within the UME associated with the mean of all averages.



3. Document any results that are outside of the acceptance criteria.
4. The final reported purity result is the mean of all accepted averages.
- E. Reports controlled substances in decreasing order of abundance (if known).
- F. Calculates and reports the quantitative result as the predominant salt form (if known).
 1. When the salt form is unknown, document the salt form used to calculate the reported quantitative value, e.g., heroin (calculated as hydrochloride).
- G. Reports the final purity result in percentage truncated to match the significance of the final reported uncertainty.
- H. Reports purity results greater than 100% (e.g., 100.6%) as 100%.
- I. Calculates and reports the purity UME per 2-7.



7.0 Determining the Uncertainty of Measurement Estimates for Quantitative Values

The FC:

- A. Calculates the uncertainty of measurement estimate (UME) associated with quantitative values per [Appendix *2D*](#).
- B. Reports the UME for the purity of all quantitated substances on the DEA-113.

7.1 Reporting UME

The FC:

- A. Calculates all UMEs using the final averaged % purity value (prior to truncation).
- B. Rounds the final expanded UME to one significant figure using ISO/NIST rounding rules:
 - 1. 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.
 - 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.
 - 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.
 - 4. 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).
- C. Documents UMEs in LIMS and on the DEA-113.
- D. 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).
- E. 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):

- A. 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:

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

8.1 Resealing Evidence: Plastic Sealed Evidence Envelopes

The FC:

- A. 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.
 - 1. For evidence not composited following Option 1, repackage evidence ***at a minimum*** in the following populations, ***as applicable*** – composite, remaining portion from analyzed units, and unanalyzed units.
 - 2. 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.
- B. Places the packaging or containers into the original evidence envelope.
- C. 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*.
- D. 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.
 - 1. Seal individual sub-exhibits in the same evidence envelope.
- E. Heat-seals the open end of the evidence envelope through the affixed seal.
- F. Inspects the integrity of the heat seal.
- G. Obtains the gross weight after analysis of each individual sealed evidence envelope.
- H. Reports the total gross weight after analysis in LIMS.
- I. 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).



- J. 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:

- A. 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.
1. For evidence not composited following Option 1, repackage evidence ***at a minimum*** in the following populations, ***as applicable*** – composite, remaining portion from analyzed units, and unanalyzed units.

- B. Reseals evidence retained in opaque containers in the following manner:

1. 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.

2. Weigh each sealed interior package or container using the balance software.
3. Record the weight and the date sealed on the package or container.
4. Document the weights in LIMS.

- C. 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.

- D. Reseals the exterior evidence container(s) with fiber-reinforced tape by completely encircling the container in two directions.

- E. 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.

- F. Obtains the gross weight of each evidence container.

- G. Reports the total gross weight after analysis in LIMS.

- H. 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).



- I. 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:

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

NOTE: It is not necessary to split units to meet the specified threshold. ****Small amounts over the threshold may be maintained, so long as the additional amount retained does not exceed the amount contained in one additional unit.****
 2. The bulk portion consists of the remaining material.
 3. ****For exhibits containing more than one substance above the respective threshold, retain based on the larger threshold amount.****
- B. Places the threshold portion and at least one empty original packaging (if possible) into a single, original or substitute evidence container.
 1. 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.
- C. Places the bulk portion and all unretained empty packaging into remaining original or substitute evidence containers.
- D. Reseals the exterior evidence containers as in 2-8.1 or 2-8.2, as appropriate.
 1. Wrap the threshold evidence container with red tape.
 2. Mark the evidence container(s) as appropriate (i.e., “threshold” or “bulk”).
- E. Selects Organize My Work, Evidence Containers.
- F. Changes the Container Code for the threshold amount to “Threshold” and the bulk portion to “Bulk Evidence.”
- G. Prints the evidence container labels with the new container code designations.
- H. Affixes the new labels over the existing container labels.



- I. Obtains the gross weight of each evidence container.
- J. Reports the total gross weight after analysis in LIMS.
- K. 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).

- L. At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- M. Includes the following statement in the *Remarks* of the *Evidence Details* section of the DEA-113:
 - 1. For DEA evidence, annotate the amount of bulk evidence, along with the intent of destruction.
“___ gram(s) held for destruction pending written notification.”
 - 2. 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:

- A. 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).
- B. Reseals the exterior evidence container(s) as in 2-8.1 or 2-8.2, as appropriate.
- C. Obtains the gross weight of each individual evidence container.
- D. Reports the total gross weight after analysis in LIMS.
- E. 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).

- F. 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:

- A. Repackages evidence using new or additional evidence containers as needed.
- B. Creates additional/new evidence container(s) in LIMS.
- C. Completes the *Lab Exhibit Description* as appropriate.
- D. Prints container labels for the newly created evidence container(s).
- E. Affixes the label(s) to the new evidence container(s).
- F. Adds the *Additional Evidence Unit* test to each newly created exhibit.



9.0 Reanalyzing Evidence

- A. 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:

- A. Reopens the exhibit in LIMS.

NOTE: Return exhibit from storage, if applicable.

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

9.1.1 Reanalysis

The FC:

- A. Makes no changes to the original analysis documentation in LIMS.
- B. Creates a new sub-exhibit in LIMS
- C. Names *Lab Exhibit* as “*Exhibit Number-R*” (e.g., 1-R, 1.01-R, 1A-K-R, 1B1-R, etc.).
- D. Documents as “*Reanalysis of Exhibit X*”, where X is the original exhibit number (e.g., 1, 1.01, 1A-K, 1B1).
- E. 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.

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



- J. Annotates the reason for reanalysis in the *Other Notes* test.
- K. Reseals the evidence as described in 2-8.1 or 2-8.2.
- L. Records a description of the reserve evidence and the date resealed in LIMS.
- M. Obtains the gross weight after analysis.
 - 1. Use a substitute code, not the original exhibit's barcode, when obtaining the gross weight after analysis.
- N. 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.
- O. At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).

9.1.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- B. 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:

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

NOTE: Return exhibit from storage, if applicable.

- C. Renames *Lab Exhibit* as “*Exhibit Number-R*” (e.g., 1-R, 1.01-R, 1A-K-R, 1B1-R, etc.).
- D. Reroutes the exhibit to Chemistry.
- E. Sends the exhibit for analysis.
- F. Assigns the exhibit to the FC.

9.2.1 Reanalysis



The FC:

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

9.2.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- B. 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

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

The SC:

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

9.3.1 Reanalysis

The FC:



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

9.3.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the reanalysis information. (See 2-11.8)
- B. 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-XXXXXX) 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:

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

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

The SC:

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

10.1.1 Removing Sample(s)

The FC:

- A. 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.
- B. Documents all observations and measurements using the *Other Notes* test in LIMS, a DEA-86 or DEA-86a.
- C. Reopens the following LIMS tests:
 - 1. Reserve Weight
 - 2. Description of Reserve Evidence



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



- L. 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.

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

10.1.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
- B. Ensures the supplemental report reflects the new reserve weight.
- C. Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:

" _____ grams removed for defense analysis."
- D. 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:

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

NOTE: Return exhibit from storage, if applicable.

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



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

10.2.1 Removing sample(s)

The FC:

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



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

10.2.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
 1. Use the appropriate non-LIMS DEA-113 on SFDCC.
- B. Adds original analysis results to the non-LIMS DEA-113.
- C. Ensures the supplemental report reflects the new reserve weight.
- D. Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:

"_____ grams removed for defense analysis."
- E. 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."
- F. Prints, signs, and submits the supplemental report to the SC.

The SC:



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

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

- A. 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 SC:

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

10.3.1 Removing sample(s)

The FC:

- A. Documents all observations and measurements using the *Other Notes* test in LIMS, the DEA-86 or DEA-86a.
- B. Adds the following LIMS tests:
 - 1. Gross Weight
 - 2. Exemplar Weight Removed
 - 3. Other Notes
 - 4. Reserve Weight
 - 5. Description of Reserve Evidence
 - 6. Gross Weight After Analysis
- C. Obtains the gross weight of the sealed evidence using the balance software.
- D. Reopens the exhibit and records the date reopened on the evidence container label.
- E. Removes, or observes the removal of, the sample(s) for defense analysis and obtains a new reserve weight of the original exhibit.



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

10.3.2 Reporting

The FC:

- A. Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 2-11.8)
 - 1. Use the appropriate non-LIMS DEA-113 on SFDCC.
- B. Adds original analysis results to non-LIMS DEA-113.
- C. Ensures the supplemental report reflects the new reserve weight.



D. Includes the following statement in the *Remarks* of the *Exhibit Details* section of the DEA-113:
" _____ grams removed for defense analysis."

E. 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."

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

The SC:

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

H. Sends non-LIMS supplemental report to case agent.

I. Attaches copy of email communication with case agent to *Case Attachments*.

10.4 Reweighing Evidence

The SC:

A. Reopens the exhibit in LIMS.

NOTE: Return exhibit from storage, if applicable.

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

C. Reroutes the exhibit to Chemistry.

D. Sends the exhibit for analysis.

E. Assigns the exhibit to the FC.

The FC:

F. Makes no changes to the original analysis documentation in LIMS.

G. Adds the *Other Notes* test to the exhibit.



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

10.5 Assessing Evidence Returned from Court

- A. Evidence returned from court shall be **processed** in accordance with LOM 73.

****10.5.1 Assessing Evidence When the Outer Seals Were Altered or Broken****

****The FC:**

- A. Makes no changes to the original analysis documentation in LIMS.
- B. Adds the *Other Notes* LIMS test.
- C. In the presence of a witness, obtains the gross weight of the evidence using the balance software.
 - 1. Use a substitute code, not the original exhibit's barcode, when obtaining the gross weight.
 - 2. Document the witness name in the *Other Notes* test in LIMS.
- D. Manually attaches the gross weight balance data to the *Other Notes* test.
- E. Reopens the exhibit and records the date reopened on the evidence container label and in the *Other Notes* test in LIMS.
- F. In the presence of a witness, visually inspects the evidence and verifies the contents against the last documented reserve evidence description.
 - 1. Document the witness name in the *Other Notes* test in LIMS.
- G. If the internal evidence seals/containers are intact:
 - 1. Reseals the evidence as described in 2-8.1 or 2-8.2.
 - 2. Records a description of the reserve evidence and the date resealed in LIMS.
 - 3. Obtains the gross weight after analysis.



- a. Use a substitute code, not the original exhibit's barcode, when obtaining the gross weight after analysis.
 4. 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.
 5. At a minimum, records the gross weight after analysis and the date resealed on the evidence label(s).
- H. If the internal evidence seals/containers are altered, notifies the supervisor and performs a reanalysis as described in 2-9.1.1.**



11.0 The Analytical Record

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

11.1 General Instructions

The FC:

- A. Uses the LIMS tests to record all raw data, observations, and calculations at the time they are made.
- B. 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.
- C. Documents the qualitative method used.
- D. Documents the quantitative method used.
- E. Documents the DEA property inventory numbers of all equipment and instruments used in the *Equipment* tab of the appropriate test.
- F. Documents the basic parameters/conditions for all instruments used in the appropriate test or on the attachments (e.g., spectra, chromatograms, etc.).
- G. Captures all weighing events using the balance software.
- H. Reports all weights, quantitation results, and uncertainties to the appropriate number of significant figures.
- I. 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.

- J. Records reference material unique identifier(s) in the appropriate test or on the attachments.
- K. ****Records traceable equipment unique identifier(s) in the appropriate test or on the attachments.****
- L. 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).
 - 1. 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:

- A. For routine analysis, adds, at a minimum, the following LIMS tests:
 - 1. Gross Weight
 - 2. Description of Evidence
 - 3. Description of Exhibit and Sampling
 - 4. Net Weight
 - 5. Reserve Weight
 - 6. Gross Weight After Analysis
 - 7. Description of Reserve Evidence
 - 8. Summary of Findings
- B. For sub-exhibit analysis, adds, at a minimum, the following LIMS tests:
 - 1. Description of Exhibit and Sampling
 - 2. Net Weight
 - 3. Reserve Weight
- C. For REDACTED Latent Print units, adds the following LIMS tests:
 - 1. Applicable SP Weight Test
 - 2. Description of Reserve Evidence
 - 3. Gross Weight After Analysis (SP/LP)
- D. For No Analysis exhibits in the possession of the FC, adds, at a minimum, the following LIMS tests:
 - 1. When seals are intact:
 - a. Gross Weight
 - b. No Analysis Performed
 - c. Summary of Findings
 - 2. When seals are broken:



- a. Gross Weight
 - b. Description of Evidence
 - c. No Analysis Performed
 - d. Description of Reserve Evidence
 - e. Gross Weight After Analysis
 - f. Summary of Findings
3. The remark “No Analysis as per [Insert Reason]” is added to the Observations, Results, and Conclusions section of the DEA-113.
- E. For exhibits re-opened, updates the following tests, as applicable:
1. Description of Evidence
 2. Reserve Weight
 3. Gross Weight After Analysis
 4. Description of Reserve Evidence
 5. Summary of Findings
- F. Completes all LIMS tests as described in [Appendix *2E*](#).

The SC:

- G. 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.
- H. Sends to storage, after status change to CR In-Processing, in the Exhibits tab of Case Management.
- I. Selects Storage Only and adds reason in the Comments field.
- J. 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:

- A. Includes spectral, chromatographic, and other instrumental data in the [LIMS case file](#).



B. Ensures that each item of data is annotated, at minimum, with:

1. A unique identifier
2. Date and time of analysis

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

The FC:

- A. Prepares a DEA-113 to report results for all analyzed evidence and proficiency testing samples.
- B. Prepares a DEA-113 to document when there was “*No Analysis Performed.*”
- C. Prepares a DEA-113 to document when there was an “*Inconclusive*” result.
 1. Use the following statement in the *Remarks* regardless of the number of units –
“**Inconclusive result;* identification pending further analysis.*” Other identified substances in the exhibit are reported per Appendix 2B.
- D. Ensures that LIMS automatically populates all fields on the DEA-113, except the *Remarks* fields.
(See 2-11.5)
- E. Reports identified substances in the following order:
 1. Controlled substances in order of abundance, if known, regardless of whether a substance was quantitated.
 - a. ****If multiple salt forms of a controlled substance are determined, report all salt forms individually in order of abundance, if known.**
 - b. **If a purity result is obtained, the salt form used to determine the purity is listed first.**

NOTE: A remark may be added stating that the reported purity is for all salt forms identified (e.g. *Exhibit contains a mixture of cocaine hydrochloride and cocaine base. The substance purity represents the total percentage of cocaine, calculated as the hydrochloride.*)**
 2. Non-controlled substances in order of abundance, if known.
- F. Reports reserve weights (non-exemplar exhibits) in the same units as the net weight and according to the following rules:
 1. 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



2. 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

3. 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

- G. Reports gross weights, net weight (exemplars), reserve weight (exemplars), separated bulk weights, REDACTED as follows:

1. Weight < 10 g – truncate and report to two significant figures (e.g., 0.86 g, 6.7 g)
2. $10 \leq \text{Weight} < 1000$ g – truncate and report to tenth of a gram (e.g., 96.2 g, 711.0 g)
3. Weight ≥ 1000 g – truncate and report to four significant figures (e.g., 2013 g, 327.6 kg)

- H. Reports volumes (net and reserve) as follows:

1. Less than 1 mL: Volume not reported.
2. Less than 100 mL: Truncate and report to one decimal place (e.g., 9.3 mL, 85.3 mL).
3. $100 \leq \text{Volume} < 1000$ mL: Truncate and report to whole (e.g., 325 mL).
4. Volume ≥ 1000 mL: Truncate and report to four significant figures (e.g., 2013 mL, 327.6 L).

- I. Report dosage units (net and reserve; tablets, capsules, impregnated paper) as follows:

1. Less than 1 dosage unit: Dosage units not reported.
2. 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).
3. 100 or more dosage units: Report to truncated whole value, if counted, calculated or extrapolated (e.g., 325 capsules).

- J. Calculates purity equivalencies as follows:

1. Tablets/Capsules: Multiply the final (truncated) % purity by the average weight per unit.
2. Liquids: Multiply the final (truncated) % purity by the density of the liquid.

- K. Truncates and reports purity equivalencies as whole numbers if result is ≥ 10 , or two significant figures if result is < 10 . For example,



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

- L. Submits completed DEA-113 for review.

11.4.1 The Laboratory Report (SFL1 only)

The FC:

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

11.5 The Laboratory Report (DEA-113) – Remarks

The FC:

- A. Utilizes standardized statements available through the “*Insert Phrase*” options in *Examiner Reports Management*.
- B. Obtains supervisory approval prior to inserting non-standardized statements.
- C. Enters statements in the *Observations, Results, and Conclusions* section to document the following, as applicable:
 - 1. Procedure for net weight determination
 - 2. Net weight uncertainty statement
 - 3. Total unit count and volume



4. Purity, when determined, and uncertainty statements
 5. Purity equivalencies, when determined (e.g., mg/unit or mg/mL)
 6. Remarks for *No analysis, Storage only, Supplemental, or Amended reports*
 7. Department of Justice (DOJ) Uniform Language for Testimony and Reports (ULTR) reference statement (refer to [Appendix 2B](#))
- D. Enters statements in the *Exhibit Details* section to document the following, as applicable:
1. REDACTED
 2. Bulk evidence separation
 3. Latent print evidence separation
 4. Packaging and gross form descriptions with an *Other* finding
 5. Explanations of abbreviations or terminology used on the DEA-113
- E. Enters statements in the *Exhibit Analysis* section to document the following, as applicable:
1. Sampling procedure for qualitative analysis (statistical or non-statistical)
 2. Qualifying statements for the reported identification(s)
- F. Enters statements in the *Certifications* section, as applicable:
1. Certificate of compliance statements

11.6 Reviewing Analysis and Laboratory Report

The FC:

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

The SC ****or designee****:

- E. Reviews the *Case Details Report*, the supporting data, and DEA-113 for technical and clerical accuracy, ensuring that:



1. Case, exhibit, and LIMS identifiers are properly documented.
 2. Gross weight and evidence descriptions are complete and consistent with the DEA-7 or equivalent.
 3. Observations and analyses are clearly and completely documented.
 4. Analytical techniques are appropriate for the sample type.
 5. Instrumental data and attachments are included and appropriately annotated (e.g., spectra, chromatograms, bulk photos).
 6. Conclusions are supported by test results.
 7. Manual calculations are accurate.
 8. All documents are free of administrative or transfer errors and improper use of abbreviations.
- F. Communicates and records any discrepancies or corrections to the analyst and returns the case electronically to the analyst for resolution.
1. For paper case files, discrepancies or corrections must be documented and included in the case file.
- G. 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.”
- H. Submits the approved report for distribution in accordance with LOM 73.

11.7 Analyzing and Reporting Foreign Drug Samples (SFL1 only)

SFL1:

- A. Analyzes enforcement REDACTED samples received from foreign offices.
- B. Reports and distributes the analytical results in accordance LOM 73.
 1. 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:



- A. Generates a supplemental DEA-113 when additional results become available or reanalysis is performed.
 1. Select the Final Report option for the Report Type.
 2. 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.”
 3. Use the *Approved By* date from the original report.
- B. Generates an amended DEA-113 when errors on the original report are corrected.
 1. Select the *Final Report* option for the *Report Type*.
 2. 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.”
 3. Use the *Approved By* date from the original report.



CHAPTER 3 – FIELD ASSISTANCE

1.0	Field Assistance	181
1.1	Scope	181
2.0	Clandestine Laboratories	182
2.1	Preparing for Clandestine Laboratory Investigations	182
2.2	On Site Activities.....	182
2.3	Analysis and Reporting	183
2.3.1	Capacity Report Memorandum.....	184
3.0	Collecting Trace Drug Evidence	186
3.1	Ion Mobility Spectrometer	186
3.1.1	Storing the IMS Equipment	186
3.1.2	Transporting the IMS Equipment.....	186
3.1.3	Setting-Up and Inspecting Equipment On-Scene	187
3.1.4	Calibrating and Maintaining Equipment On-Scene.....	187
3.1.5	Collection Filters	187
3.2	Collecting Evidence	187
3.3	Submitting Evidence	189
3.4	Upon Return to Laboratory.....	189
3.5	Reporting Results	189
4.0	Processing Synthetic Drugs.....	190
4.1	Plant Material.....	190
4.1.1	Hazardous Materials	190
4.1.2	Non-Hazardous Materials	190
4.2	Powder Chemicals	190
4.3	Retail Packages	190
4.3.1	Same Brand and Same Flavor	190
4.3.2	Same Brand and Different Flavors	191
4.3.3	Different Brands.....	191



4.4	Liquids	191
4.5	Sampling Table	192



1.0 Field Assistance

1.1 Scope

- A. 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).
- B. Laboratory personnel use the procedures described in this chapter, in conjunction with the REDACTED.



2.0 Clandestine Laboratories

The Laboratory Director (LD) or designee:

- A. Coordinates all clandestine laboratory responses within the laboratory's area of responsibility in which DEA asserts primary authority.
- B. 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:

- A. Briefs Special Agents (SAs) on technical matters pertinent to the investigation upon request.
- B. 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.
- C. 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.
- D. 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:

- A. Enters the laboratory only after the premises are secured by SAs or TFOs.
- B. 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.
- C. 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.
 - 1. 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.
 - 2. The SA or TFO must be present and document any communication with the REDACTED, operating personnel, or members of the press.
- D. Directs the shut-down of all operational equipment, if applicable, after determining the manufacturing sequence.



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

REDACTED

- G. Assists SAs or TFOs with debriefing suspected operating personnel involved in the investigation, and obtains information of a technical nature.

1. REDACTED

- H. 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.
- I. Photographs and/or records all essential areas of the clandestine laboratory, as well as exhibits seized. (See 2-2.5)
- J. Performs field tests on site, when applicable.
- K. Documents items seized as evidence with unique identifying information. Ensures that the items can be recognized in court.
- L. Assists SAs or TFOs in identifying solvents and other hazardous materials present at the laboratory site for proper disposal by hazardous waste contractors. REDACTED
- M. 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:

- A. 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:

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



- E. After completing the analysis of clandestine laboratory evidence in which there was not a participating DEA FC, prepares a DEA-500 upon request only.
- F. 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.
- G. Offers expert opinions at trial regarding estimated actual yields, or upon receiving a written request from the prosecutor.

The SC:

- H. Reviews the DEA-500.
- I. Stamps all copies of the completed DEA-500 "*DEA Sensitive*."
- J. Attaches the DEA-500 in [LIMS](#) to *Case Attachments* as attachment type DEA-500.
- K. REDACTED
- L. Sends copies of the report to the following offices:
 - 1. The office head or the designee of the office conducting the investigation
 - 2. 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)
 - 3. REDACTED
 - 4. Drug and Chemical Evaluation Section (DPE), Headquarters
 - 5. Synthetic Drugs and Chemicals Section (DOS), Headquarters
- M. Forwards a copy of the transmittal letter(s) to the Office of Forensic Sciences.

2.3.1 Capacity Report Memorandum

The FC:

- A. 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.
 - 1. 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:



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



3.0 Collecting Trace Drug Evidence

The LD or designee:

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

The FC:

- C. Accompanies the SAs, TFOs, or Diversion Investigators (DIs) to conduct a trace evidence collection/vacuum search for drug evidence.
- D. 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:

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

3.1.2 Transporting the IMS Equipment

The FC:

- A. Inspects and ensures that the IMS is operational, prior to deploying it for field operation.
- B. Transports the IMS in a travel crate.
- C. If the equipment is being shipped:
 - 1. Labels the outside of the travel crates as "FRAGILE."
 - 2. 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.
- D. Locks the crate(s), if permitted by the shipping company or airline.



- E. Transports the IMS via air cargo, if permitted by airline regulations.

NOTE: The same declaration of radioactive materials is required.

- F. Hand-carries the computer.

3.1.3 Setting-Up and Inspecting Equipment On-Scene

The FC:

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

3.1.4 Calibrating and Maintaining Equipment On-Scene

The FC:

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

3.1.5 Collection Filters

The FC:

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

3.2 Collecting Evidence

The FC:

- A. Collects samples as follows:



1. Blank all filters to be used on the IMS.
 - a. 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.
 - b. 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.
 - a. Vacuum the air near the IMS instrument, and check the sample on the IMS.
 - b. Save the plasmagram on the computer hard drive, so that the data can be linked back to the environmental blank.
 - c. 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.
 - a. If there was a positive IMS result, vacuum the same area more thoroughly to obtain a “heavy” sample.
 - b. 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.
- B. 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:

- A. 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:

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

3.5 Reporting Results

The FC:

- A. 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:

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

The FC:

- B. 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

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

4.1.2 Non-Hazardous Materials

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

4.2 Powder Chemicals

- A. Submit all material to the laboratory.

4.3 Retail Packages

4.3.1 Same Brand and Same Flavor

- A. Under 2 kg of material inside packets:
 - 1. Submit all packets as a single exhibit.
- B. Over 2 kg of material inside packets:
 - 1. 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.



2. 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

- A. Under 2 kg of material inside packets:

1. Submit all packets.

- B. Over 2 kg of material inside packets:

1. 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.
2. 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

- A. Under 2 kg of material inside packets:

1. Submit all packets of each brand as separate exhibits (e.g., exhibits “1,” “2,” “3,” etc.).

- B. Over 2 kg of material inside packets:

1. 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.
2. 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

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



- C. 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

CHAPTER 4 – FINGERPRINT REDACTED

1.0 Fingerprint Program.....	195
1.1 Separating and Preserving DEA Fingerprint Evidence	195
1.2 Preserving Fingerprint Evidence Not Separated from Drug Evidence	196
1.3 Separating Fingerprint Evidence for Other Agencies.....	196
1.4 Sampling for Fingerprint Examination of Bulk Drug Evidence Seizures.....	196
1.5 Drug Evidence Packaging - Bench Transfers.....	197
REDACTED	198
REDACTED.....	198
REDACTED.....	199
REDACTED.....	201
REDACTED	202
REDACTED.....	202
REDACTED	202
REDACTED	204
REDACTED.....	204
REDACTED.....	206
REDACTED.....	206
REDACTED	207
REDACTED.....	207
REDACTED.....	208
REDACTED.....	209
REDACTED	210
REDACTED.....	210
REDACTED	212
REDACTED.....	212
REDACTED.....	212
REDACTED.....	214

REDACTED215
REDACTED..... 215

1.0 Fingerprint Program

1.1 Separating and Preserving DEA Fingerprint Evidence

The FC:

- A. Carefully separates all packaging from the alleged controlled drug substance, removing as much controlled drug substance as possible, leaving little or no residue.
- B. While conducting the separation, handles the **physical evidence** carefully to preserve any latent prints that might be present.
- C. Places the separated fingerprint evidence into an additional container (e.g., plastic bag).
 - 1. Ensures that any drug paraphernalia is packaged to prevent accidental injury (e.g., covers exposed hypodermic needles, packages razor blades separately, etc.).
- D. Marks the additional container with the **LIMS case number**, initials, and date before inserting the separated fingerprint evidence.
- E. Places additional container into an evidence container.
- F. Creates a Fingerprint Unit (FIN) in LIMS ****and adds the LIMS tests listed in 2-11.2.****
- G. ****Completes the following lines on the evidence label:**
 - 1. **CASE NUMBER** – enter Investigating Agency (IA) case number
 - 2. **EXHIBIT NUMBER** – enter laboratory exhibit number
 - 3. **SEALED BY** – FC prints name, signs, and dates**
- H. Seals and annotates the evidence container (e.g., Latent Print Examination).
- I. 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.
 - 1. For evidence sent to another laboratory for latent print examination, includes a statement in the transmittal documents identifying the potential hazard.
- J. ****Obtains the gross weight of the PSEE, and records the weight on the envelope label.****
- K. Returns the fingerprint evidence to the vault.
- L. 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 prints cannot be removed without destroying latent prints which may be present (e.g., LSD blotter papers).

The FC:

- A. Contacts the submitting agent to determine if both the drug analysis and the fingerprint processing are needed.
- B. Contacts a Fingerprint Specialist (FS) to determine the best way to handle and process the evidence if both examinations are needed.
- C. Annotates the evidence container according to 4-1.1.

1.3 Separating Fingerprint Evidence for Other Agencies

The FC:

- A. 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.

- B. Carefully separates all packaging from the alleged controlled drug substance as in 4-1.1.
- C. Creates additional units in LIMS.
 - 1. Create Fingerprint Units (FIN) for containers being returned to the other agency for latent print examination. Route to CT.
 - 2. Follow procedures in 4-1.1 for evidence being analyzed by DEA. Route to CT/LP with SC approval.
- D. 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:

- A. Determines fingerprint examination procedures for **bulk** seizures (i.e., the number of units to be examined), in consultation with the FC, Supervisory Fingerprint Specialist, FS, and an appropriate enforcement official on a case-by-case basis.

1.5 Drug Evidence Packaging - Bench Transfers

The FC:

- A. Follows the bench transfer procedure below when the latent print examination will be conducted while the evidence is in the possession of the FC:
 1. 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.
 2. Transfer the evidence to the FS in LIMS.
 3. After fingerprint analysis and reassignment of the exhibit, receive the evidence from the FS in LIMS.
- B. Upon completion of the fingerprint and chemical analyses, reseals both the drug and original fingerprint evidence.
- C. Annotates the *Remarks* in the *Exhibit Details* section of the DEA-113: “*Original evidence submitted for latent print examination.*”

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Appendix 1A – Definitions

Term	Definition
Accuracy	The closeness of agreement between the experimental value and the true value.
Adulterant	A non-controlled but pharmacologically active substance that may be added to a controlled substance.
Analogue (21 U.S.C. § 802)	(A) Except as provided in subparagraph (B), the term "controlled substance analogue" means a substance: (i) the chemical structure of which is substantially similar to the chemical structure of a controlled substance in schedule I or II; (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 (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. (B) Such term does not include: (i) a controlled substance; (ii) any substance for which there is an approved new drug application; (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 (iv) any substance to the extent not intended for human consumption before such an exemption takes effect with respect to that substance.
Analytical Balance	A balance with a readability between 0.1 mg and 0.01 mg (i.e. 4-place and 5-place).

Term	Definition
Analytical Scheme	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.
Bias	The difference between the expectation of the test results and an accepted reference value.
Biohazard	Infectious agents or hazardous biological materials that present a risk or potential risk to the health of humans.
Bulk exhibit	Contraband drug evidence submitted to a DEA laboratory whose net weight exceeds the threshold amount .
Bulk portion	Amount of contraband drug evidence in excess of the appropriate threshold amount .
**Calibrant	A traceable solution prepared from a certified reference material and used to establish a calibration curve, either by itself or along with additional calibrants.
Calibration Curve	The mathematical relationship that exists between the analyte concentration or sample amount and the signal, over a selected range of concentrations.**
Case Details Report (CDR)	The summary of analytical testing within LIMS.
Certified reference material (CRM) (ISO Guide 30:2015*(E))	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. **SFL1-produced CRMs have traceable purity values of 98% or greater and an expanded uncertainty of no greater than 1.25%.**
Co-analysis	Comparison of an analyte and reference material in the same experiment by simultaneous analysis.
Combined standard uncertainty (u)	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 .

Term	Definition
Composite	Representative, homogenized material prepared in accordance with the Evidence Sampling Plan (ADM 2-4) and the sampling procedures in Appendix 2C .
Confirmation technique	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.
Confirmed	See Reporting Terms
Correlated measurements	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.
Coverage factor	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> .
Critical resolution pair	For separation analyses , a pair of compounds eluting or migrating with a baseline resolution between 1.5 and 5.0 at half-height.
Determined	Use of one test to obtain information (e.g., salt form, purity, isomer). For use on DEA-113, see Reporting Terms.
Diluent	A substance typically used to increase the bulk of a finished product.
Exemplar	A submitted portion of a larger seizure that may or may not be representative of the entire seizure.
Exhibit	Physical evidence submitted to the laboratory. See also Sub-exhibit.
Expanded uncertainty (<i>U</i>)	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.
Gummy exhibit	Exhibit which is not amenable to grinding or mixing.
Identified	See Reporting Terms.
Increment	Randomly chosen portion from the exhibit from which the composite is assembled.
Instrument blank	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.

Term	Definition
Investigating Agency (IA)	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.
Laboratory exhibit number	A LIMS specific field, which directly relates to the IA exhibit number, used to designate laboratory created sub-exhibits.
Limit of Detection (LOD)	An estimate of the lowest analyte concentration or sample amount that can be reliably differentiated from the blank matrix.
Limit of Quantitation (LOQ)	An estimate of the analyte concentration or sample amount that can be reliably measured with acceptable precision (repeatability) and accuracy (bias).
LIMS	Laboratory information management system
LIMS case file	Electronic record of all actions performed on a piece of evidence. The record may include the items found in LOM 73.
LIMS case number	A unique identifier which refers to a single IA exhibit .
Linearity	The ability of a method to produce test results that are directly proportional to analyte concentration within a given range.
Low-response compound	Compound that produces a low-intensity signal under routine experimental conditions.
Measurand (VIM, 3rd Ed.)	Quantity intended to be measured. • Net weight - The measured weight of an exhibit. • Purity - The measured fraction of an exhibit associated with the identified substance. • Amount of Pure Drug - The calculated amount of actual identified substance in an exhibit defined by the net weight multiplied by the purity.
Measurement assurance (ASCLD/LAB-International AL-PD-3059)	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.
Measurement uncertainty	Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand.

Term	Definition
Method	The combination of the technique (separation, confirmation) and associated operating parameters required for an analysis. **General purpose – Applicable to the analysis of a wide range of substances including dimethyl sulfone, methamphetamine, phenyltetrahydroimidazothiazole (PTHIT), cocaine, heroin, oxycodone, fentanyl, and trazodone. Limited purpose – Applicable to a group of substances as defined within the scope of the method.**
Method validation	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.
Method verification	An assessment whether a validated method performs as expected under actual conditions of use (i.e. transfer to a new laboratory or new instrument).
Negative control (blank)	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.
Orthogonal techniques (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.
Physical evidence	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.
Positive control	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.
Presumptive test	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 .
Probe technique	A procedure whereby units are pierced or small openings are made and a small amount of material is removed for analysis.

Term	Definition
Procedural blank	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.
Quality control (QC) sample	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.
Quality control (QC) solution	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.
Quality control (QC) check	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.
Reagent ** (IUPAC Gold Book)**	*A test substance that is added to a system in order to bring about a reaction or to see whether a reaction occurs.*
Reference compound	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.
Reference material (RM)	A homogeneous, stable, and traceable material which has been established to be fit for use in identification or quantitation of substances.
Relative area	The ratio of the peak area of one compound relative to the peak area of a reference compound .
Relative retention / migration time	The ratio of the elution (retention/migration time) of one compound relative to the elution (retention/migration time) of a reference compound .
Relative standard deviation (RSD)	For replicate measurements, the measured standard deviation divided by the mean.
Repeatability	The degree of agreement among individual test results when the procedure is applied multiple times, over a short time interval.
Representative Sample	Exemplar for testing and a sample aggregate portion of the whole amount seized sufficient for current criminal evidentiary practice.

Term	Definition
Reporting terms:	
Identified or Confirmed	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).
Determined	Used for reporting results for salt form, optical isomer, and purity.
Reproducibility	The degree of agreement among individual test results when the procedure is applied multiple times, over an extended time interval.
Residue	A quantity of substance for which the determination of a weight is not practical or the weight is less than *15* mg.
Reviewable data	Information obtained from analytical methodology or documents containing recorded forensic observations.
Ruggedness	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.
Selectivity	The separation of the analyte(s) of interest and the internal standard, if utilized, from other sample components in a mixture/matrix.
Selectivity solution	Method-specified solution containing the target analyte and additional components at concentrations commonly encountered in laboratory submissions.
Separation technique	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.
Standard uncertainty	Measurement uncertainty expressed as a standard deviation.
Sub-exhibit	The separation of an exhibit resulting from significantly different chemical composition, color, appearance, etc.
Target analyte(s)	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.
Technique	Wet chemical or instrumental tests that provide information about the composition of a substance. Examples include mass spectrometry, infrared spectroscopy, or color tests.
Test portion	The amount withdrawn for qualitative or quantitative analysis.

Term	Definition
Threshold amount ** (28 CFR §50.21) **	The required size of a representative sample from a bulk exhibit involving the following Schedule I and II Controlled Substances: • Heroin: 2 kg of a mixture or substance containing a detectable amount of heroin. • Cocaine: 10 kg of a mixture or substance containing a detectable amount of: ○ Coca leaves, except coca leaves and extracts of coca leaves from which cocaine, ecgonine, and derivatives of ecgonine or their salts have been removed. ○ Cocaine, its salts, optical and geometric isomers, and salts of isomers. ○ Ecgonine, its derivatives, their salts, isomers, and salts of isomers. ○ Any compound, mixture, or preparation which contains any quantity of any of the substances referred to in the preceding three bullet points. • Cocaine base: 10 kg of a mixture or substance containing cocaine base.
(Continued below)	

Term	Definition
Threshold amount (Continued)	• PCP: 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). • LSD: 20 g of a mixture or substance containing a detectable amount of Lysergic Acid Diethylamide (LSD). • Fentanyl: 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]. • Fentanyl analogues: 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>. • Hashish: 20 kg of hashish or two kilograms of hashish oil [21 USC 841(b)(1)(D), 960(b)(4)]. • Other Schedule I or II: 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. • Marijuana: 10 kg of a mixture or substance containing a detectable amount of marijuana. 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.
Traceability (VIM, 3 rd Ed., 2.41)	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.

Term	Definition
Unique identifier	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. For weighing events, the unique identifier is either the container ID, the universal weight ID, or the test ID barcode. For reference materials, the identification number that provides traceability.
Uncorrelated measurements	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.
Working range	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.



Appendix 1B – Acronyms and Abbreviations

- A. Listed below are the acronyms and abbreviations for use by DEA laboratory personnel. The acronyms and abbreviations are not case sensitive.
- B. Acronyms and abbreviations defined in the following references are also approved for use.
1. American Chemical Society (ACS) Style Guide
 2. Chemical Abstracts Services (CAS) Standard Abbreviations and Acronyms Listing B
 3. Diversion Control Division - Controlled Substance Schedules
 4. Laboratory Operations Manual (LOM)
 5. Merck Index
 6. Official Methods of Analysis of AOAC International
 7. SWGDRUG Drug Monographs
- C. Abbreviations for the latent print program can be found in the Latent Print Examination Manual (LPEM), while abbreviations for the digital evidence program can be found in the Digital Evidence Examination Manual (DEEM).
- D. 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.
- E. Any abbreviations not listed below or in the above references must be defined in the case details report upon first use.

Compounds	
6MAM	O6-Monoacetylmorphine
AC	Acetylcodeine
AMPH	Amphetamine
APAP	Acetaminophen
BICARB	Bicarbonate
BMPEA	α -benzyl-N-methyl- β -phenethylamine
C13	Tridecane
C20	Eicosane
C22	Docosane
C24	Tetracosane



Compounds	
CAFF	Caffeine
CBC	Cannabichromene
CBD	Cannabidiol
CBG	Cannabigerol
CBN	Cannabinol
COC	Cocaine
DMSO2	Dimethyl Sulfone
DMTP	Dimethylterephthalate
EPHED	Ephedrine
HER	Heroin
METH	Methamphetamine
MJ	Marijuana
NN-DMA	N,N-Dimethylamphetamine
OXY	Oxycodone
PDMAB	para-Dimethylaminobenzaldehyde
PDMAC	para-Dimethylaminocinnamaldehyde
PSEUDO	Pseudoephedrine
PTHIT	Phenyltetrahydroimidazothiazole
8-THC	Delta-8-Tetrahydrocannabinol
THC	Delta-9-Tetrahydrocannabinol

Evidence Descriptions	
BC	Body Carry
Cap	Capsule
CKB	Compressed Kilo Brick
CM	Crystalline Material
KB	Kilo Brick
Plt Mat	Plant Material
Tab	Tablet

Instrumentation	
APCI	Atmospheric Pressure Chemical Ionization



Instrumentation	
CE	Capillary Electrophoresis
DART	Direct Analysis in Real Time
DESI	Desorption Electrospray Ionization
IMS	Ion Mobility Spectrometry
LTM	Low Thermal Mass
QTOF	Quadrupole Time of Flight
UPLC	Ultra Performance Liquid Chromatography

LIMS	
CDR	Case Details Report
CIF	Combined Instrument Files
CR	Central Receiving
CT	Chemical Testing
IA	Investigative Agency
LASS	Laboratory Activity Summary Sheet
SDMS	Scientific Data Management System
SO	Storage Only
REDACTED	REDACTED

Miscellaneous	
Alkd	Alkaloid
ATM	Adulterant Test Mixture
B/C	Because
Bioh	Biohazard
Bkgd	Background
BI	Blank
Clan Lab	Clandestine Laboratory
Comp	Composite
REDACTED	REDACTED
Decon	Decontamination
DNMC	Does Not Meet Criteria
Eff	Effervescence
ESP	Evidence Sampling Plan



Miscellaneous	
REDACTED	REDACTED
Ex	Exhibit
GWAA	Gross Weight After Analysis
GW	Gross Weight
Haz	Hazardous
HQC	High Quality Control Sample
REDACTED	REDACTED
Id	Identification
Inconcl	Inconclusive
Ind	Indicated
INO	Insolubles Not Observed
IO	Insolubles Observed
IS	Internal Standard
ISS	Internal Standard Solution
LQC	Low Quality Control Sample
Macro	Macroscopic exam
Micro	Microscopic exam
Mod	Modified
REDACTED	REDACTED
MT	Migration Time
NCS	No Controlled Substance
NCSD	No Controlled Substance Detected
NFA	No Further Action
NW	Net Weight
Orig	Original
PBI	Procedural Blank
QNS	Quantity Not Sufficient
QS	Quantity Sufficient
Qual	Qualitative
Quant	Quantitative
Repst	Representative
Rgt	Reagent
RM	Reference Material
RMT	Relative Migration Time



Miscellaneous	
RT	Retention Time
RTV	Return to Vault
RW	Reserve Weight
Rxn	Reaction
SC	Supervisory Chemist
Scrn	Screen
SFC	Senior Forensic Chemist
Sol	Soluble
Spl	Sample
SU	Salt Form Undetermined
TW	Tare Weight
Und	Undetermined
Unk	Unknown
W/	With
W/O	Without
Subst	Substitute

Packaging	
BT	Black Tape
CB	Carbon Paper
Cello	Cellophane
CKPB	Clear Knotted Plastic Bag
CP	Clear Plastic
CPB	Clear Plastic Bag
CPW	Clear Plastic Wrap
CZPB	Clear Zip-Lock Plastic Bag
EE	Evidence Envelope
Env	Envelope
GB	Glassine Bag
GT	Grey Tape
HS	Heat Sealed
KPB	Knotted Plastic Bag
NZPB	New Ziplock Plastic Bag
OZPB	Original Ziplock Plastic bag



Packaging	
PB	Plastic Bag
PKG	Package
PSB	Plastic Sandwich Bag
PSEE	Plastic Sealed Evidence Envelope
PW	Plastic Wrap
SSEE	Self-Sealing Evidence Envelope
TT	Tan Tape
VSB	Vacuum Seal Bag
WPB	Whirl Pack Bag
ZPB	Ziplock Plastic Bag

Reagents	
BSA	N,O-Bis(Trimethylsilyl) Acetamide
BSTFA	N,O-Bis(Trimethylsilyl) Trifluoroacetamide
CoSCN	Cobalt Thiocyanate
DIPC	Diisopropylcarbodiimide
DuqL	Duquenois-Levine
MRQ	Marquis Reagent
M Scott	Modified Scott's Reagent
MSTFA	N-Methyl-N-(Trimethylsilyl) Trifluoroacetamide
MTPA	alpha-Methoxy-Alpha-Trifluoromethylphenylacetic Acid
MTPA-CI	alpha-Methoxy-Alpha-Trifluoromethylphenylacetyl Chloride
NaNP	Sodium Nitroprusside
PIT	Phenylisothiocyanate
TDTA	di-p-Toluoyl-(d/l) Tartaric Acid
TFAA	Trifluoroacetic Acid
TPC	N-Trifluoroacetyl-L-Prolyl-Chloride

Solvents	
SolvA-0.01	0.01 mg/mL C24 in 4:1 Chloroform: Methanol
SolvA-0.0125	0.0125 mg/mL C24 in 4:1 Chloroform: Methanol
SolvA-0.05	0.05 mg/mL C24 in 4:1 Chloroform: Methanol
SolvB-0.01	0.01 mg/mL C24 in Ammonia-Saturated Chloroform



Solvents	
SolvB-0.0125	0.0125 mg/mL C24 in Ammonia-Saturated Chloroform
SolvC-0.0125	0.0125 mg/mL C24 in Chloroform with 10% Sodium Hydroxide
SolvD	0.4 mg/mL C24 in 9:1 Chloroform: Methanol
THC-ISS-A	0.05 mg/mL 4-androsten-3,17-dione in 9:1 MeOH:Chloroform
THC-ISS-T	0.05 mg/mL testosterone in 9:1 MeOH:Chloroform
β-CD	beta-Cyclodextrin
ACN	Acetonitrile
AF 3.7	10mM Ammonium Formate Buffer, pH 3.7
AMH	Ammoniacal Hexane
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

****The frequency of instrument checks stated in this section are minimum requirements. Checks should be performed if system performance deteriorates.****

1 Capillary Electrophoresis

Frequency of Check	Parameter	Procedure
Date of operation	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.
Monthly	Detector	Perform Diode Array Detector test. Replace the lamp(s) when necessary.
Monthly	Electrodes and pre-punchers	Check and clean as needed.
Every 3 months	Pressure system	Examine the inlet and outlet seals. Replace the air filter if applicable.

2 Liquid Chromatograph

Frequency of Check	Parameter	Procedure
Monthly	Detector	Using the column eluent, perform a diode array detector intensity check for max and min levels and perform a wavelength calibration.
Every 2 years	Cleanliness	Perform a check of the system. Check and clean or replace solvent inlet filters, buffer reservoirs, as needed.
Every 2 years	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	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	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	Source (Electrospray)	Clean spray chamber and capillary spray shield. Replace if necessary.
Every 3 months	Source (APCI)	Clean corona needle if in use. Replace after 3 months of use or earlier if necessary.
Every 6 months	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	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.
According to manufacturer's specifications	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.

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.

****16 Portable Mass Spectrometer**

Frequency of Check	Parameter	Procedure
If high background warning received	Trace Module	Inspect and clean according to manufacturer's instructions.
If clean cycle fails after cleaning trace module	Mass Spec Core	Inspect and clean according to manufacturer's instructions.
After replacement	Mass Spec Core	Run calibration.**

****17 Automatic Pipettes**

Frequency of Check	Parameter	Procedure
Every 6 months	Calibration	Calibrate semi-annually by an ISO/IEC 17025-accredited calibration laboratory.**

****18 Electronic Temperature Monitoring System**

Frequency of Check	Parameter	Procedure
Yearly	Calibration	Calibrate temperature sensor annually.



Appendix *1D* – Qualitative **and Quantitative** 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:

1. Lower the split ratio.
2. Change in the sample solvent (except NMR).
3. Increase the injection volume.
4. Shorten the solvent delay.
5. Extend the hold time at the end of a method.
6. Increase the number of scans (IR, Raman, and NMR only).
7. 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:

1. Trimming of a column.
2. 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.

3. The method check includes:
 - a. A single injection of the repeatability mixture from validation (1-3.1.2.1)
 - b. 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.



4. Documentation of the check, results, *and technical review* in the instrument logbook
- C. ****The following modifications to quantitative methods are permitted without further action:**
 1. Extend the hold time after the established run time of the validated method.
 2. Change in gradient (i.e., temperature, buffer ratio, voltage, flow) after the established run time of the validated method.
- D. **The following modifications to quantitative methods require a check of the method:**
 1. Replacement of a column with another one having the same properties and dimensions.
 2. The method check includes:
 - a. Analysis of the critical resolution pairs.
 - b. Analysis of one injection of the QC-high and the QC-low solutions.
 - c. Evaluation of the data per acceptance criteria (1-4).
 - d. Documentation of the check, results, and technical review in the instrument logbook.
- E. **Reducing the run time of a validated qualitative or quantitative method requires a re-evaluation of validation parameters in coordination with SF.****
- F. **The following changes to qualitative ****and quantitative methods**** result in a new method and require a new method name and complete validation:**
 1. Change in gradient (i.e., temperature, buffer ratio, voltage, flow).
 2. Change in detector parameters (i.e., scan rate on MS, resolution on IR).
 3. Installation of a column with different properties, dimensions, or technology.
 4. Implementation of a different buffer composition (e.g., phosphate buffer changed to acetate buffer or pH 3 changed to pH 5).
 5. Change in the scan range.
 6. Change in carrier gas ****for separatory methods.****

NOTE: For instrument ***maintenance*** and required performance verification procedures see 1-6.



Appendix *1E* – Analytical Supplies and Services

Providers (vendors) of analytical supplies and services must be evaluated to ensure they meet the requirements listed below.

Consumables		
Item(s)	Vendor Requirement(s)	Additional Notes
Reference Materials	Accreditation through: ISO/IEC 17025 ISO 17034	
Services		
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
Automatic Pipette Calibration	Accreditation through ISO/IEC 17025	Required every 6 months
Electronic Thermostat Calibration	Accreditation through ISO/IEC 17025	Required annually**
Traceable Glassware Calibration	Accreditation through ISO/IEC 17025	Required every 10 years**
Supplies		
External Proficiency Samples	Accreditation through ISO/IEC 17043	
Reagents		See 1-11



Appendix 2A – Random Sampling Procedures

- A. Random sampling procedures are required when selecting units from an exhibit (i.e., the population) for net weight determination, qualitative analysis, and composite formation.
- B. Random selection processes are used to ensure:
 - 1. All units in a population have an equal chance of being selected.
 - 2. Selection **bias** is avoided.
- C. Random selection:
 - 1. Allows the use of statistical methods to analyze sample results.
 - 2. Allows inferences to be made on the population.
- D. **Method 1:** Random number generator (RNG)
 - 1. Arrange or stack all population units in a pattern.
 - 2. Open the **RNG** and enter the total number of units in the exhibit and the number of units to be selected.
 - 3. Identify the units to be sampled by following a row or a column starting at the upper left corner.
 - 4. Segregate or label (if possible) the units selected (unit 1, unit 2, unit 3, etc.)
- E. **Method 2:** Lottery Method A
 - 1. Place units into one or more containers (bowls, bags, etc.).
 - 2. Mix the units thoroughly.
- OR
- 3. Randomly arrange or stack all population units in a pattern (columns, rows, or both).
 - 4. Perform 'blind' selection by removing one unit at a time.
 - 5. Segregate or label (if possible) the units selected (unit 1, unit 2, unit 3, etc.).
- F. **Method 3:** Lottery Method B
 - 1. Arrange or stack all population units in a pattern (columns, rows, or both).
 - 2. Place numbered pieces of paper (or balls, marbles, etc.) in a container (bowl, bag, etc.).



3. Mix the numbers thoroughly.
4. Perform 'blind' selection by reaching into the container and removing one piece of paper (or ball, marble, etc.) at a time.
5. 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.
6. 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.

Liquids and Dosage Units (additional remark)



Analysis of Drugs Manual
Appendix 2B – Reporting Statements for Net Weight Determination,
Identification(s), and Purity Result(s)

Revision: 5

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Net Weight Determination Statements for Remarks Section of DEA-113 form:

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: One or more substances confirmed in all units tested and additional controlled substance(s) confirmed in some but not all units tested.

****2-9 units:** [List Substance(s) Identified] confirmed in [#] units tested of [#] units received. [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}.

****10 or more units:**** [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



Analysis of Drugs Manual
Appendix 2B – Reporting Statements for Net Weight Determination,
Identification(s), and Purity Result(s)

Revision: 5

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Identification Statements for Remarks Section of DEA-113 form:

[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}*.

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.

****Data insufficient for confirmation in the pre-composite, confirmation in the composite:** *[#]* units tested of *[#]* units received. A composite was formed from *[#]* units for further testing. *[List Substance(s) Identified]* confirmed in the composite. Salt form [and/or] isomer determined from testing 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}*.

****Single unit (composite formed):** X.01 A composite was formed from 1 unit for testing. *[List Substance(s) Identified]* confirmed in the composite. Salt form [and/or] isomer determined from testing the composite.**

X.02 No analysis per *[Enter approval source]*. ****Conclusions reported for X.01 cannot be applied to the unanalyzed units in X.02.****

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.



Analysis of Drugs Manual
Appendix 2B – Reporting Statements for Net Weight Determination,
Identification(s), and Purity Result(s)

Revision: 5

Issue Date: March 22, 2021

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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

A. If practical, the FC forms the composite by combining the entire contents of all units in the exhibit (including untested units, if present).

1. 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.

B. 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.

C. For liquids, the resulting composite is mixed thoroughly.

Option 2: Performing incremental sampling

A. Powders, crystalline materials, and body carries

1. Units containing at least 1 g of material:

- a. One increment is approximately 1 g.
- b. Form **approximately a** 15-g composite.
- c. 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.

- d. Combine all increments to form the composite.
- e. 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.



2. Units containing less than 1 g of material:

- a. One increment = one unit.
- b. 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.
- c. 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**).

- d. Combine all increments to form the composite.
- e. 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.

3. Units containing both < 1 g and > 1 g of material:

- a. 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.
- b. 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.
- c. 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.

- d. Combine all increments to form the composite.
- e. 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.

B. Liquids and Solutions

1. One increment = 1 mL (or entire unit if < 1 mL)
2. 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.

3. Combine all increments to form the composite.
4. Mix thoroughly.

C. Solid Dosage Forms

1. One increment = One dosage unit or one partially remaining unit after testing
2. 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**).

3. Combine all increments to form the composite.
4. Grind, sieve to a maximum particle size of 850 μm (20-mesh), and mix thoroughly.



Appendix *2D* – 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.

$$u_{mass} = \sqrt{u_{bal}^2 + u_{process}^2 + u_{acc}^{**2}} \quad \text{Equation 1}$$

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>u_{mass} (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 processes²

² A static weighing process involves two separate weighing events, the weighing of the vessel by itself and the weighing of the vessel with material.



and correlated measurements; therefore, all combined net weight uncertainties are calculated by 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:

$$\text{Average NW} \pm u_{NW} \quad \text{where, } u_{NW} = \sqrt{u_{mass}^2 + u_{avg}^2}$$

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.
 $u_{RM} = 0.005774$

****The traceability of purity measurements is established through the use of certified reference materials, analytical balances calibrated to traceable reference standards, and certified Class-A glassware or calibrated pipettes. Measurement assurance is provided by contemporaneous analysis of quality control (QC) samples per 2-6.4. The DEA property inventory numbers of all instruments used are documented in LIMS. Combined and expanded (95% level of confidence) uncertainty calculations are performed within the LIMS *Summary of Findings* test, and the final absolute uncertainty results included in the case file and laboratory report (DEA-113).****

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 collaborators³⁴ 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.

$$RSD = 0.0716 - 0.012\ln(\%P) \quad \text{Equation 7}$$

³ 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.

⁴ Boyer KW, Horwitz W, Albert R. Interlaboratory variability in trace element analysis. Anal. Chem. 1985; 57:454-459.

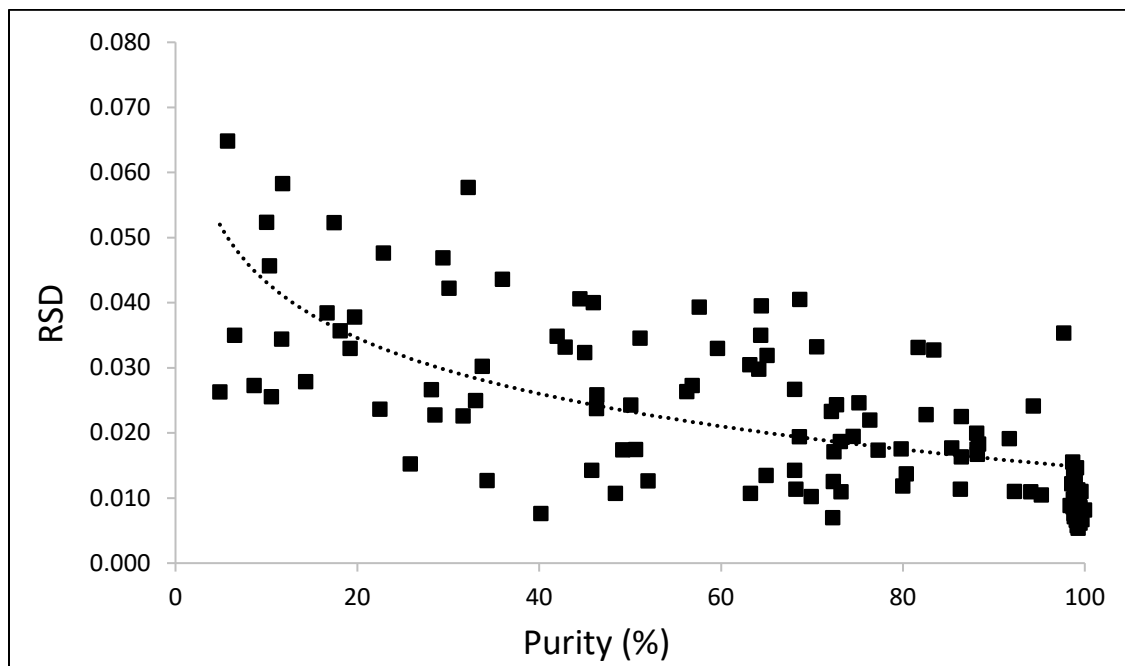


Figure 1: Dependence of PTP RSD values on purity

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.

$$u_{acc} = 0.1215 - 0.021\ln(\%P)$$

Equation 8

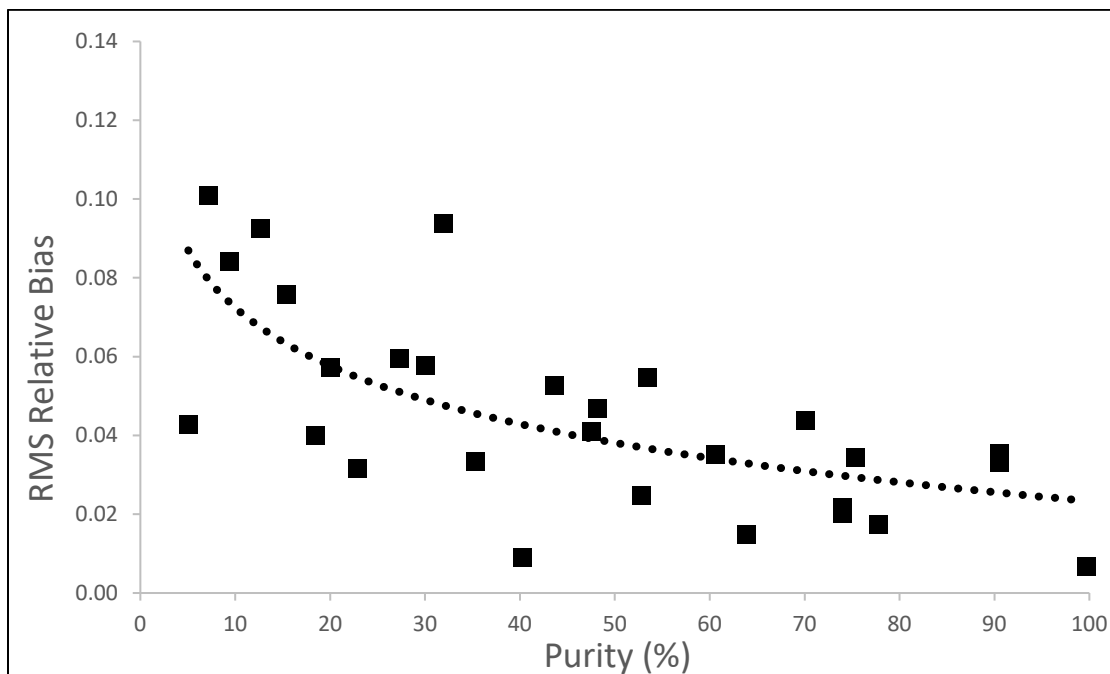


Figure 2: Dependence of RMS relative bias on concentration for PTP samples prepared by SFL1 (2014-2018)

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:

$$APS \pm U_{APS} = APS \pm (APS) (u'_{APS}) (k) = APS \pm (APS) \left(\sqrt{(u'_{NW})^2 + (u'_p)^2} \right) (k)$$

where,

u'_{APS} , u'_{NW} , and u'_p are the relative uncertainties associated with amount of pure substance, net weight, and purity, such that

$$u'_{APS} = \left(\frac{u_{APS}}{APS} \right) = \left(\sqrt{(u'_{NW})^2 + (u'_p)^2} \right) \text{ and } u'_{NW} = \left(\frac{u_{NW}}{NW} \right) \text{ and } u'_p = \left(\frac{u_p}{P} \right)$$

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 *2E* – Instructions for Completing LIMS Tests

1	Gross Weight Test	261
2	Description of Evidence Test	261
3	Description of Exhibit and Sampling Test.....	261
4	Net Weight Test	263
5	Net Weight (Sub-Group) Test	263
6	Net Weight (Exemplar) Test.....	264
7	Composite Weight Test.....	264
8	Additional Evidence Unit Test	264
9	No Analysis Performed Test	264
10	Logo Identification.....	265
11	pH Test	265
12	Solubility/Miscibility Test	265
13	Watesmo Paper Record Test.....	266
14	Macro/Microscopic Examination of Plant Material	266
15	Microscopic Examination Test	266
16	Color Tests	267
17	**Precipitate Tests**	267
18	*Microcrystalline* Tests	268
19	Immunoassay Tests	269
20	Thin Layer Chromatography Test.....	269
21	Chromatographic Tests.....	270
22	Hyphenated Tests.....	270
23	NMR Test	271
24	Vibrational Spectroscopy Tests.....	271
25	UV/Vis Test.....	272
26	IMS Test	272
27	Polarimetry Test.....	273
28	Deviation Test.....	273



29	Quantitation Test	273
30	Other Notes Test	275
31	REDACTED	275
32	Exemplar Weight Removed Test.....	275
33	REDACTED	275
34	REDACTED	275
35	REDACTED	275
36	REDACTED	276
37	REDACTED	276
38	Gross Weight – REDACTED/Latent Print (REDACTED/LP) Test.....	276
39	Reserve Weight Test	276
40	Reserve Net Weight (Exemplar) Test.....	277
41	Reserve Weight (Direct Bulk) Test	277
42	Reserve Weight (Bulk) Test	278
43	Description of Reserve Evidence Test	278
44	Gross Weight After Analysis Test.....	278
45	Supervisory Approval Test	279
46	Weight/Volume PTP Test	279
47	SFL1 Only Tests	279
47.1	Enforcement Exhibit Test	280
47.2	Enforcement Exhibit Test	280
47.3	REDACTED	280
48	Summary of Findings Test	281



1 Gross Weight Test

- A. Weigh the sealed evidence as received from the vault using the balance software.
- B. Ensure the *Gross Weight (Actual)* finding is populated correctly.
 - 1. For sub-exhibits, the *Gross Weight* test is added only to the first sub-exhibit.
- C. Obtain a witness for the *Weight Discrepancy* finding, if needed.
- D. Document the balance used in the *Equipment* tab.

2 Description of Evidence Test

- A. For sub-exhibits, the *Description of Evidence* test is added only to the first sub-exhibit.
- B. Record the condition of the seals as received in the *Seals* finding.
 - 1. Obtain a witness in the *Seal Witness* finding, if the seals are not intact.
- C. Enter the date the evidence was opened in the *Date Opened* finding.
 - 1. If the evidence was opened more than once, annotate the *Remarks* finding with each opening date.
- D. 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.
 - 1. 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."
- E. Select "Yes/No" in the *Consistent with Paperwork* finding as appropriate.
- F. Obtain a witness to any discrepancy.
 - 1. 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

- A. Add the Description of Exhibit and Sampling test to all sub-exhibits.
 - 1. Describe the packaging in the first sub-exhibit when the sub-exhibits are submitted in one container.



2. Set the *Package Type* finding to “*Described in first exhibit split*” for all of the remaining sub-exhibits.
- B. Enter the number of innermost packages containing the suspected controlled substance in the *Number of Packages* finding.
- C. Enter the total number of units in the exhibit in the *Number of Units* finding.
 1. For commingled residue exhibits, enter the *Number of Units* as 1.
- D. Select the appropriate descriptions for the innermost packaging and the contents of the exhibit in the *Package Type* finding.
 1. Select “*Other*” when the exhibit contains multiple package types or when the package type is not listed.
 - a. The packaging shall be described in the *Remarks* finding.
 - b. Include a statement describing the packaging in the *Exhibit Details* section on the DEA-113.
- E. Set the *Logo/Impression* finding to “*Yes*” if a submission to *Operation Fountainhead* is made.
- F. Select the appropriate form of the material to be analyzed in the *Gross Form* finding.
 1. Select “*Other*” when the exhibit contains different forms or when the form is not listed.
 - a. The gross form shall be described in the *Remarks* section.
 - b. Include a statement describing the form(s) in the *Exhibit Details* section on the DEA-113.
- G. Select “*Dry*”, “*Moist*” or “*N/A*”, as applicable.
- H. Set the *Exemplar* finding to “*Yes*” if the sample is an exemplar exhibit.
- I. Enter the number of units analyzed in the *Number of Units Tested* finding.
- J. 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).
- K. Obtain supervisory approval for deviations from the evidence sampling plan (ESP).
 1. The supervisor electronically approves the deviation in the *Deviation Approved By* finding before the analysis is completed.



2. Documents the reason for the deviation in the *Reason for Deviation* finding.

L. All pictures of the exhibit not related to another specific LIMS test shall be attached to the *Image* finding.

4 Net Weight Test

A. Select “Yes/No” in the *Residue* finding, as appropriate.

B. Select the type of weighing performed in the *Type of Weighing* finding.

C. Obtain the net weight of the exhibit using the balance software.

D. For liquids and solutions: use the balance software to determine the density of the composite and calculate the total volume of the exhibit.

E. For solid dosage forms: determine the total number of units by counting or by extrapolation using the balance software.

F. 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.

G. 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).

H. Ensure the net weight and the net weight uncertainty populate the *Net Weight* test fields correctly.

I. Document the use of the *Legacy Calculator* and obtain supervisory approval of the deviation.

J. Document the balance used in the *Equipment* tab.

5 Net Weight (Sub-Group) Test

A. 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).

B. 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.

C. Ensure the net weight and the net weight uncertainty populate the *Net Weight* test fields correctly.

D. Document the use of the *Legacy Calculator* and obtain supervisory approval of the deviation.

E. Document the balance used in the *Equipment* tab.



6 Net Weight (Exemplar) Test

- A. Use this test only for exemplar exhibits.
- B. Select the type of weighing performed in the *Type of Weighing* finding.
 - 1. Use the *Exemplar Different A* balance method for exhibits composed of one representative and multiple core-type samples.
 - 2. Use the *Exemplar Same Container* balance method for all other types of exhibits.
- C. Enter the total number of units in the *Number of Units* finding.
- D. Enter the net weight in the *Total Net Weight of Exhibit* finding.
- E. 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.
- F. Document the balance used in the *Equipment* tab.

7 Composite Weight Test

- A. Use this test as needed.
- B. Set the *Reserve Composite Weighed* finding to “Yes” or “No”, as applicable.
- C. Enter the initial composite weight and reserve composite weight in the appropriate findings.
- D. Describe the item(s) used to obtain the tare weight (e.g., packaging, substitute packaging, etc.) in the *Remarks* finding.
- E. Document the balance used in the *Equipment* tab.

8 Additional Evidence Unit Test

- A. 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.).
- B. Add the phrase “*All tests listed in Unit 1*” in the *Remarks* finding.

9 No Analysis Performed Test

- A. Enter “No Analysis Performed” in the *Results* finding.
- B. 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

- A. This test is used for information only and cannot be used to fulfill the minimum identification requirements of two tests/two portions.
- B. Record the logo identification information (i.e., 5 mg Oxycodone, 650 mg Acetaminophen) in the *Logo ID* finding.
- C. Record the source used to make the logo identification in the *Logo ID Source* finding.
 - 1. 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.
 - 2. If the source is not listed in the *Logo ID Source* finding, select "Other" and record the source used in the *Remarks* finding.
- D. If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- E. Make an annotation as to which units were tested in the *Remarks* finding.
 - 1. 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

- A. Record the method used to make the pH measurement in the *Method of pH Measurement* finding.
- B. Record the pH measured in the *Measured pH* finding.

12 Solubility/Miscibility Test

- A. Select either the *Solubility* or *Miscibility* finding.
- B. Record the solvent used in the *Solvent* finding.
- C. Select the appropriate value for the *Miscibility Result* finding, if tested.
- D. Select the appropriate value for the *Solubility Result* finding, if tested.
- E. If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- F. 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

- A. Select the appropriate value in the *Color Observed* finding.
- B. If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- C. 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

- A. Record the macroscopic description of the plant material in the *Macroscopic Observation* finding.
 - 1. Select “*Other*” for the *Macroscopic Observations* finding to enter an explanation in the *Remarks* finding.
- B. Enter the magnification used to examine the plant material in the *Magnification* finding.
- C. Record all observations made while performing the microscopic examination in the *Microscopic Observations* finding.
 - 1. Select “*Other*” for the *Microscopic Observations* finding to enter an explanation in the *Remarks* finding.
- D. Document the microscope(s) used in the *Equipment* tab.
- E. If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- F. 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

- A. 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.



- B. Enter the magnification used to examine the material in the *Magnification* finding.
- C. Record all observations made while performing the microscopic examination in the *Microscopic Observations* finding.
- D. Document the microscope(s) used in the *Equipment* tab.
- E. If the same result is obtained for all samples tested, document it under a single run/set (e.g., Run 1, Set 1).
- F. 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

- A. Uses this procedure for all color tests.
- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
 - 1. Record the Negative Control Result finding as “Pass” or “Fail.”
 - 2. ****Record the lot number or unique ID of the reagent(s) in the Reagent ID finding.****
- C. Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
 - 1. Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
 - 2. ****Record the lot number or unique ID of the reagent(s) in the Reagent ID finding.****
 - 3. Select the appropriate color observed from the values in the *Color* finding.
 - 4. If no matching color exists in the drop-down menu, select “Other” and record the color observed in the *Remarks* finding.
- D. Annotate which units were tested and give the same results in the ***Applicable Units*** finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used.

17 ****Precipitate Tests****

- A. **Use this procedure for all precipitate tests.**



- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
1. Record the Negative Control Result finding as “Pass” or “Fail.”
 2. **Record the lot number or unique ID of the reagent(s) in the Reagent ID finding.
- C. Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
1. Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
 2. **Record the lot number or unique ID of the reagent(s) in the *Reagent ID* finding.
 3. Enter the observations in the *Precipitate Observed* finding.
- D. Annotate which units were tested and give the same results in the **Applicable Units** finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used.

***18* *Microcrystalline* Tests**

- A. Use this procedure for **all microcrystalline tests.**
- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
1. Record the Negative Control Result finding as “Pass” or “Fail.”
 2. **Record the lot number or unique ID of the reagent(s) in the Reagent ID finding.**
- C. Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
1. Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
 2. **Record the lot number or unique ID of the reagent(s) in the *Reagent ID* finding.**
 3. Enter the observations in the *Crystals Observed* finding.
- D. Annotate which units were tested and give the same results in the **Applicable Units** finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used.



****19 Immunoassay Tests****

- A. ****Use this procedure for all immunoassay tests.**
- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
 - 1. Record the Negative Control Result finding as “Pass” or “Fail.”
 - 2. Record the type of immunoassay detection in the Immunoassay Type finding.
 - 3. Record the lot number of the strips in the Lot Number finding.
- C. Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
 - 1. Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
 - 2. Record the lot number of the strips in the *Lot Number* finding.
 - 3. Enter the observations in the *Results* finding.
- D. Annotate which units were tested and give the same results in the *Applicable Units* finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used.**

***20* Thin Layer Chromatography Test**

- A. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
 - 1. Record the Negative Control Result finding as “Pass” or “Fail.”
- B. Use the second set of each run to document the result of the samples by setting the *Negative Control Run* finding to “No.”
 - 1. Use a single run/set (e.g., Run 1, Set 1) if the same result is obtained for all samples tested.
- C. 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.
 - 1. 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.
- D. Record the name(s) of the compound(s), reference materials, and retention factor(s) into the *Results* finding.



- E. Annotate which units were tested and give the same results in the **Applicable Units** finding (e.g., “Result applies to Units 1–29”).

NOTE: The annotation “x29” is not self-documenting and therefore may not be used.

***21* Chromatographic Tests**

- A. Use this procedure for the following tests: *CE Analysis*, *HPLC Analysis*, and *GC Analysis*.
- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
1. Select the appropriate value for the *Negative Control Type* finding.
 2. Record the *Negative Control Result* finding as “Pass” or “Fail.”
- C. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
1. Indicate if the sample was weighed in the *Sample Weighed* finding.
 2. Enter the appropriate values into the *Sample Prep* findings.
- D. Record the solvent used to dissolve the sample (e.g., CHCl_3 , sample dissolved in H_2O and basified with 1.0 N $\text{NaOH}_{(\text{aq})}$ and extracted into CH_2Cl_2 , etc.) in the *Solvent* finding.
- E. 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.
- F. Document the instrument(s) and balance(s) used in the *Equipment* tab.

***22* Hyphenated Tests**

- A. Use this procedure for the following tests: *GC-IRD Analysis*, *GC-MS Analysis*, and *LC-MS Analysis*.
- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
1. Select the appropriate value for the *Negative Control Type* finding.
 2. Record the *Negative Control Result* finding as “Pass” or “Fail”.
- C. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
1. Indicate if the sample was weighed in the *Sample Weighed* finding.



2. Enter the appropriate values into the *Sample Prep* findings.
- D. Record the solvent used to dissolve the sample (e.g., CHCl_3 , Sample dissolved in H_2O and basified with 1.0 N $\text{NaOH}_{(\text{aq})}$ and extracted into CH_2Cl_2 , etc.) in the *Solvent* finding.
- E. Set the *Retention Time Matching* finding to “No” for spectral results only.
 1. Add the name(s) of the compound(s) identified to the *Spectral Result* finding.
 2. If the separation test (retention times) is used to fulfill the identification requirements, the results must be documented.
- F. Set the *Retention Time* finding to “Yes” for the instrument to perform retention time calculations.
 1. Record the name(s) of the compound(s), reference material, retention time(s), and corresponding area counts in the *Chromatographic Results* finding.
- G. Document the instrument(s) and balance(s) used in the *Equipment* tab.

***23* NMR Test**

- A. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
 1. Select the appropriate value for the *Negative Control Type* finding.
 2. Record the *Negative Control Result* finding as “Pass” or “Fail”.
- B. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
 1. Indicate if the sample was weighed in the *Sample Weighed* finding.
 2. Enter the sample weight and dilution volume into the *Sample Weight* and *Final Dilution Volume* findings, respectively.
- C. Select the solvent used for the *Solvent* finding.
 1. If no matching solvent exists in the drop-down menu, select “Other” and record the solvent used in the *Remarks* finding.
- D. Enter the name(s) of the compound(s) identified into the *Results* finding.
- E. Document the instrument(s) and balance(s) used in the *Equipment* tab.

***24* Vibrational Spectroscopy Tests**

- A. Use this procedure for the following tests: *FTIR Analysis* and *Raman Analysis*.



- B. Use the first set of each run to document the negative control test by setting the *Negative Control Run* finding to “Yes.”
 - 1. Select the appropriate value for the *Negative Control Type* finding.
 - 2. Record the *Negative Control Result* finding as “Pass” or “Fail.”
- C. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
 - 1. Enter the sample preparation used (e.g., Direct, CHCl₃ Solubles, Acetone Insolubles/CHCl₃ Solubles, etc.) in *Sample Prep* finding.
- D. Enter the name(s) of the compound(s) identified into the *Results* finding.
- E. Indicate the instrument(s) used in the *Equipment* tab.

***25* UV/Vis Test**

- A. Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
 - 1. Record the *Negative Control Result* finding as “Pass” or “Fail.”
- B. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
- C. Enter the name(s) of the compound(s) identified into the *Results* finding.
- D. Document the instrument(s) used in the *Equipment* tab.

***26* IMS Test**

- A. Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
 - 1. Select the appropriate value for the *Negative Control Type* finding.
 - 2. Record the *Negative Control Result* finding as “Pass” or “Fail.”
- B. Record the result for each sample that corresponds to a specific negative control in the same run using subsequent sets.
- C. Set the *Verification Test* to “Yes” if the sample is a verification sample.
- D. Record the *Verification Test Result*: “Pass” or “Fail.”
- E. Record the name of the compound(s) identified in the *Results* finding.



F. Document the instrument(s) used in the *Equipment* tab.

***27* Polarimetry Test**

- A. Use the first set of each run to document the negative control test by setting the *Negative Control* finding to “Yes.”
 - 1. Select the appropriate value for the *Negative Control Type* finding.
 - 2. Record the *Negative Control Result* finding as “Pass” or “Fail.”
- B. Use subsequent sets to document individual sample results by setting the *Negative Control Run* finding to “No.”
- C. Enter the observed optical rotation in the *Observed Rotation* finding.
- D. Document the instrument(s) used in the *Equipment* tab.

***28* Deviation Test**

- A. Describe the deviation in the *Description of Deviation* finding.
- B. Documents approval of the deviation in the *Deviation Approved By* finding.

***29* Quantitation Test**

- A. Select “Sample,” “Standard,” “Check,” or “Blank” in the *Type* finding.
- B. Indicate the method name/number and technique used (i.e., DEA101/Gas Chromatography, COC-LC/Liquid Chromatography, etc.).
- C. Provide details of any modification to the validated method in the *Remarks* finding.
 - 1. Obtains supervisory approval in the *Deviation* test.
- D. Documents the type of dilution in the *Dilution Technique* finding, if applicable.
 - 1. For *Volumetric*, *Gravimetric*, and *Volumetric/Gravimetric* dilutions, complete all applicable sample preparation findings.
- E. Enter the appropriate amount in the *Sample Amount (Instrument)* finding.
 - 1. For the reference material, use the purity-corrected concentration or weight of the calibrant.
 - 2. 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%.



- F. Enter the appropriate factor in the *Dilution Factor* finding.
- G. Document the calibrant(s) and QC solution preparations in the *Remarks* finding or as an attachment. Include the following information:
1. Name, salt form, and lot number/identifier of the reference material or QC sample used.
 2. ****Traceable unique identifier(s) associated with calibrated equipment used for calibrant solution preparation.****
 3. Weight, volume, dilution, final concentrations, and preparations date(s).
- H. Document the instrument(s) and balance(s) used in the *Equipment* tab.
- I. 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 ₄	1.0737
	HCl → Base	0.7879
	SO ₄ → HCl	0.9313
	SO ₄ → Base	0.7338
	Base → HCl	1.2692
	Base → SO ₄	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.



***30* Other Notes Test**

- A. This test is used to record any relevant procedures, testing, or observations not captured in other LIMS tests.

***31* REDACTED**

- A. REDACTED
 - 1. REDACTED
- B. REDACTED
- C. REDACTED
- D. REDACTED

***32* Exemplar Weight Removed Test**

- A. Add this test to the parent exhibit.
- B. Enter the total amount of material removed for REDACTED.

***33* REDACTED**

- A. REDACTED
- B. REDACTED
- C. REDACTED
- D. REDACTED

***34* REDACTED**

- A. REDACTED
- B. REDACTED
- C. REDACTED

***35* REDACTED**

- A. REDACTED
- B. REDACTED
- C. REDACTED



D. REDACTED

***36* REDACTED**

A. REDACTED

B. REDACTED

C. REDACTED

***37* REDACTED**

A. REDACTED

REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

E. REDACTED

***38* Gross Weight – REDACTED /Latent Print (REDACTED /LP) Test**

- A. Add this test to an exhibit that qualifies for a REDACTED sampling, Latent Print examination, or defense analysis sample (DFA).
- B. Indicate the weight of the sealed REDACTED, *Latent Print*, or *DFA* evidence.
- C. Document the balance used in the *Equipment* tab.

***39* Reserve Weight Test**

- A. Select “Yes” or “No” in the *Residue* finding.
- B. 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).
- C. Enter the final reserve weight of exhibit in the *Reserve Weight* finding.
 - 1. Reserve weight is reported using the same units as the reported net weight.
 - 2. For bulk exhibits, the reserve weight is the entire remaining amount including threshold and bulk portions.



- D. For liquid exhibits, enter the total reserve volume in the *Reserve Volume* finding.
- E. For dosage units, enter total reserve unit count in the *Reserve Dosage Units* finding.
- F. 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.
- G. Document the balance used in the *Equipment* tab.

***40* Reserve Net Weight (Exemplar) Test**

- A. Select the type of weighing performed in the *Type of Weighing* finding.
 - 1. Use the *Reserve Exemplar Different A* balance method for exhibits composed of one representative and multiple core-type samples.
 - 2. Use the *Reserve Exemplar Same Container* balance method for all other types of exhibits.
- B. Enter the total number of units in the *Number of Units* finding.
- C. Enter the final reserve weight in the *Total Reserve Net Weight of Exhibit* finding.
 - 1. Reserve weight is reported using the same units as the reported net weight.
- D. 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.
- E. Document the balance used in the *Equipment* tab.

***41* Reserve Weight (Direct Bulk) Test**

- A. This test is used to document the amount separated for threshold and bulk, when the *Reserve Weight (Bulk)* test is not used.
- B. Use this test in conjunction with the *Reserve Weight* test.
 - 1. For DHS bulk exhibits in which the threshold and bulk portions are not separated, the bulk reserve weights tests should not be utilized.
- C. Enter the total reserve weight in the *Total Reserve Net Weight* finding.
- D. Enter the amount of the exhibit to be retained in the *Threshold Weight* finding.
- E. Enter the amount of the exhibit to be destroyed in the *Bulk Weight* finding.



***42* Reserve Weight (Bulk) Test**

- A. This test may be used to obtain the reserve weight of a bulk exhibit when the net weight was obtained by extrapolation.
- B. Use this test in conjunction with the *Net Weight*, *Composite Weight*, and *Exemplar Weight Removed* tests.
 - 1. For DHS bulk exhibits in which the threshold and bulk portions are not separated, the bulk reserve weights tests should not be utilized.
- C. 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).
- D. Indicates the initial net weight, amount of composite used, and the amount of exhibit removed for REDACTED in the corresponding findings.
- E. Indicates the number of units placed in the threshold container for the *Total Units in Threshold* finding.
- F. Populates the *Average Weight per Container* finding from the *Uncertainty Calculator*.
- G. Calculates the *Threshold Weight* and *Bulk Weight* findings.

***43* Description of Reserve Evidence Test**

- A. Describe all reserve evidence items in the *Description* finding.
 - 1. For sub-exhibits, add this test to the first unit and include a description of each sub-exhibit.
 - 2. 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.”

- B. Record the date the container was sealed in the *Date Sealed* finding.
- C. If applicable, document each instance the container(s) was resealed in the *Remarks* finding.
- D. ****If applicable, document threshold or interior packaging weights in Case Attachments.****

***44* Gross Weight After Analysis Test**

- A. Record the weight of the sealed evidence after analysis.
 - 1. For sub-exhibits, the *Gross Weight After Analysis* test is added only to the first sub-exhibit.



- B. Indicates the balance used in the *Equipment* tab.

***45* Supervisory Approval Test**

- A. In the *Description of Action Taken* finding, describe the scenario or action requiring supervisory approval.
- B. Documents approval obtained in the *Action Approved* finding, before the analysis is completed.

****46 Weight/Volume PTP Test****

- A. ****Select *Weight* or *Volume* in the *PTP Type* finding.**
- B. **Select *Not Applicable* from the substance list in the *Result* finding.**
- C. **For PTP Type *Volume*, enter the room temperature in the *Room Temperature* finding.****

47 SFL1 Only Tests

- A. For sub-exhibit scenarios, add this test to each sub-exhibit.
- B. For the *Gross Weight* findings of sub-exhibits (e.g., X.02, X.03, etc.), enter “N/A”.
- C. For residue scenarios, net and reserve weights that are residue, enter “0.001g” in the *Net Weight* and *Reserve Weight* findings.
- D. 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*”.)
- E. When no quantitation is performed, complete the *Quantitation* finding with “N/A”.
- F. Report quantitative analysis results as directed in [Appendix *2E*-47*](#)
- G. If no analysis is performed, add the appropriate test and select “*No Analysis Performed*” as the *Qualitative* finding.
- H. Complete all other findings with “N/A”.
- I. REDACTED
 - 1. REDACTED.
- †REDACTED.
- ‡REDACTED.



***47.1* REDACTED**

- A. REDACTED
- B. REDACTED
- C. REDACTED
- D. REDACTED
- E. REDACTED
- F. REDACTED
- G. REDACTED
- H. REDACTED
- I. REDACTED
- J. REDACTED

***47.2* REDACTED**

- A. REDACTED

***47.3* REDACTED**

- A. REDACTED
- B. REDACTED
- C. REDACTED
- D. REDACTED
- E. REDACTED
- F. REDACTED
- G. REDACTED
- H. REDACTED.
- I. REDACTED



***48* Summary of Findings Test**

- A. This test is used to report conclusions from the exhibit's analysis.
- B. Add this test to the first sub-exhibit only, but includes the conclusions for all sub-exhibits.
- C. 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.*

- 1. *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.

- 2. *Substance Purity:* Report purity and UME as percent.
- 3. *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.

- 4. *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)			
Expected Purity (%):	Test amounts (mg) ^a for composites ground, mixed, and sieved to:		
	850 μm (20-mesh):	425 μm (40-mesh):	250 μm (60-mesh):
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	*>15 ^b *
35	394	50	
40	323	41	
45	267	34	
50	221	28	
55	183	23	
60	151	19	
65	124	16	
70	100	*>15 ^b *	
75	79		
80	60		
85	43		
90	28		



Powders, Crystalline Materials, and Solid Dosage Forms (NW ≥ 100 mg)			
Expected Purity (%):	Test amounts (mg) ^a for composites ground, mixed, and sieved to:		
	850 µm (20-mesh):	425 µm (40-mesh):	250 µm (60-mesh):
95	*>15 ^b *		
100			

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 *5-place balance minimum* weight determinations.



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End of Document

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