DRUG ENFORCEMENT ADMINISTRATION

LABORATORY OPERATIONS MANUAL
ANALYSIS OF DRUG EVIDENCE
7500

Contents

7501 QUALITY ASSURANCE

7501.1 Scope

The quality assurance program for drug analysis ensures reported results are reliable and includes the following:

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. Use of suitable externally provided products and services
H. Reagent reliability
I. Peer review

7501.2 Definitions

A. Terminology used in 7500 is defined in Exhibit 1/7501 Definitions
B. Use of abbreviations is defined in Exhibit 2/7501

7502 PROFICIENCY TESTING PROGRAM

7502.1 Schedule of Proficiency Samples

A. Inter-laboratory proficiency testing program (PTP) samples are prepared and distributed by DEA laboratories according to the following schedule:

Laboratory	Month
SFL1	February, April, June, August, October, December
SFL2	March, July, November
SFL3	February, June, October
SFL4	January, May, September
SFL5	April, August, December
SFL6	March, July, November
SFL7	February, June, October
SFL8	January, May, September

B. Weight and volume PTP samples (W/V-PTP) are prepared by the Office of Forensic Sciences Quality Assurance Section (SFQ) once per accreditation cycle.

C. Internal (intra-laboratory) proficiency testing program (IPTP) samples are prepared by DEA laboratories as needed to meet Laboratory Operations Manual (LOM) 7100 requirements.

D. Samples reanalyzed for purposes other than proficiency testing (i.e., court, inspection) may satisfy the annual proficiency testing requirement for an analyst provided the IPTP acceptance criteria are met per 7502.4.

E. External proficiency testing program (EPTP) samples are obtained from an ISO/IEC 17043 accredited provider per location per fiscal year.

F. Blind proficiency testing program (BPTP) samples are prepared by SFL1 in coordination with SFQ and the Office of Inspections (IN) every fiscal year.

7502.2 Preparing, Packaging, and Distributing Proficiency Samples

A. SFL1 prepares PTP samples of known composition to be used in the evaluation of purity uncertainty of measurement estimates according to the SFL1 Standard Operating Procedure (SOP).

7502.2.1 Preparing PTP and IPTP Samples from Evidence

The Laboratory Quality Assurance Manager (LQAM) or designee:

A. Selects evidence that meets the criteria in LOM 7400.

1. For PTPs, ensure a minimum of 1.0 g for each laboratory and 5.0 g for the source is available.

2. For IPTPs, ensure a minimum of 1.0 g for the sample and 1.0 g for the source is available.

B. Follows PRO-7403.2A Converting Evidence to Laboratory Program Stockpiles.

C. Documents the PTP sample preparation process, including all weights, preparer's name and preparation date.

D. Homogenizes the composite, passes the resulting material through a sieve (60-mesh for PTPs and the same mesh size as the original analyst for IPTPs), and mixes thoroughly.

1. For PTPs, when there is no composite material or there is insufficient material in the composite portion for distribution, prepare a composite from the bulk material according to Exhibit 7/7524.

a. Analyze at least one sampling of the prepared composite prior to distribution by performing, at a minimum, a confirmation test and a separation test using appropriate techniques and validated methods.

NOTE: Hyphenated techniques may be used for this purpose.

7502.2.2 Preparing Weight and Volume PTPs

The Office of Forensic Sciences Quality Assurance Manager (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.

7502.2.3 Packaging of PTP and IPTP Samples

The LQAM, SFQAM, or designee:

A. Places the testing sample(s) into the required number of containers.

1. PTP: Place at least a 1.0 g portion from the prepared bulk material into an individual vial for each laboratory location and SFT.
2. W/V-PTP: Prepare individual PSEEs for each vial.
3. IPTP: Place a at least a 1.0 g portion from the composite into a vial.
4. Place remaining bulk material (source) into a separate container (e.g. Zip-lock bag).

B. Labels each sample and source container with the proficiency test number.

1. PTP: Use the format: PTP-YY-MM-L (e.g., PTP-23-01-4 for a FY23 January sample prepared by SFL4)
   a. L is the originating laboratory number designator
2. W/V-PTP: Use the format: PTP-X-YY
   a. X is W for weight PTP and V for volume PTPs.
3. IPTP: Use the format: IPTP-YY-NN
   a. NN is a sequential count of the number of IPTPs issued during the fiscal year (e.g. IPTP-23-01 for the first FY23 IPTP at a particular laboratory).
4. Seals each individual sample and source material, if applicable, into separate new PSEEs labeled with the proficiency test number.
   a. For weight and volume PTPs, annotate the PSEE with a statement clearly specifying weight or volume measurement only, no chemical analysis.

7502.2.4 Distributing PTP Samples

The LQAM or designee:

A. For PTP samples sent to other laboratories,

1. Prepares DEA-12 forms annotated with the gross weight of the sample.
2. For W/V-PTPs, includes an instruction memorandum with the following details to ensure analysis consistency:
   a. Only weight (and volume, if applicable) results are reported.
   b. “Not Applicable” is reported in the Substance(s) Identified table on the DEA-113
   c. The remark “Analyzed for weight (and volume) measurement only” is included in the *Exhibit Analysis* section on the DEA-113.
3. Ships samples by the fifth business day of the month scheduled.
4. Emails the SFT Section Chief or designee the same day the PTP sample is shipped, alerting the training facility to expect an inbound delivery.

B. For PTP and IPTP samples remaining at the originating location, submits sample and source material to an evidence specialist via a DEA-12 annotated with the gross weight of each sample.

The SFT Section Chief or designee:

C. Manages the use of PTP samples received.

D. Designates the samples as either proficiency tests for the training staff or for general training purposes.

1. W/V-PTP samples are designated as proficiency tests for the training staff.

7502.3 PTP Sample Deadlines and Posting of Results

The LQAM or designee:

A. Ensures the analysis is complete by the following deadlines:

1. PTP:

a. Completed results are due by the 10th business day of the month following sample receipt.

2. W/V-PTP:

a. Analysis must be complete within 30 calendar days following sample receipt.

b. Results are due within 10 business days following analysis completion.

B. Posts the analysis results for PTPs and W/V-PTPs as follows:

1. PTP:

a. DEA-113

b. The Forensic Chemist Worksheet (DEA-86) or Case Details Report (CDR)

c. Combined instrumental files

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

2. W/V-PTP:

a. DEA-113

b. The Forensic Chemist Worksheet (DEA-86) or CDR

c. Associated balance data files

7502.4 Evaluation of Results

7502.4.1 Evaluating Qualitative Results

The LQAM at the originating laboratory or designee:

A. Reviews the results and identifies any qualitative inconsistencies. A qualitative inconsistency exists if any of the following conditions are met:

1. PTPs:

a. A laboratory reports a controlled substance, NPS or non-controlled substance not corroborated by at least one other laboratory.

b. A laboratory does not report or indicate in the case file a controlled substance or NPS identified by at least five other laboratories, including the original analysis.

c. A laboratory does not report a controlled substance or NPS known to be present or indicate in the case file a non-controlled substance known to be present in a PTP sample of known composition.

2. IPTPs:

a. A controlled substance or NPS is reported in only one of the two analyses.

7502.4.2 Evaluating Quantitative Results

The LQAM at the originating laboratory or designee:

A. Evaluates the results as follows, and identifies any quantitative inconsistencies.

1. PTPs:

a. Using the associated data files, calculate purity values from each laboratory truncated to one decimal place.

b. Using the PTP Summary blank form posted to the Office of Forensic Sciences Document Control Center (SFDCC), subject 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. Update the equation in the blank form to use the Grubbs statistic corresponding to the number of laboratories reporting a quantitation value.

NOTE: The ESD outlier test requires a minimum of three values.

Number of Observations	Grubbs Statistic (T_n)
9	2.21
8	2.13
7	2.02
6	1.89
5	1.72
4	1.48
3	1.15

d. Declare an outlier when T_n exceeds the Grubbs statistic value for the corresponding number of observations ($T_{critical}$ at 95% confidence, two-sided test).

NOTE: The blank form will generate a "fail" value for any outliers that meet this criterion provided the equation is updated to the appropriate Grubbs statistic.

e. Remove a single outlier from the sample data and recalculates the experimental mean, even when the outlier falls within the target range.

i. In the event that additional outliers appear, forward the summary report to SFQ. SFQ will make the final determination whether to include or remove any analytical results before calculating the mean and will notify laboratories of the results.

f. Establish 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.

g. Identify an inconsistency when a quantitative value is determined to be an outlier and falls outside the target range.

2. IPTPs:

a. 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.

7502.4.3 Evaluating Weight or Volume Results

The SFQAM or designee:

A. Evaluates the Weight and Volume PTP results and identifies any inconsistencies.

B. Ensures all weight measurements are reported to the same precision.

C. Ensures all volume measurements are calculated by density determination, per 7523.

D. 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.

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

E. 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).

7502.5 Summary of Results

7502.5.1 PTPs

The LQAM at the originating laboratory or designee:

A. Prepares a summary report of the results using the PTP Summary spreadsheet on the SFDCC.

B. Posts the summary report and disseminates to the LQAMs and SFQ by the fifth business day of the month following receipt of results.

The SFQAM or designee:

C. Reviews the results of PTPs for additional process improvement opportunities.

7502.5.2 IPTPs

The LQAM or designee:

A. Reviews the results of the IPTPs for additional process improvement opportunities.

B. Provides information to the LD regarding the results of analysis and documentation of follow-up action for inclusion in the laboratory's annual management review.

7502.5.3 W/V-PTPs

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 LQAMs and SFQ by the fifth business day of the month following posting of laboratory results.

C. Includes the results and destruction authorization on the quarterly PTP summary report.

7502.6 Documentation

The LQAM or designee:

A. Maintains documentation in the proficiency sample source exhibit (e.g. PTP-YY-MM-L Exhibit 2) in LIMS related to each proficiency sample originated and analyzed.

1. PTP and W/V PTP

a. The originating laboratory/office retains the following:

i. PTP sample number

ii. Sample preparation information

iii. The results and data of the qualitative testing, when applicable

iv. For PTPs prepared from evidence, a copy of the completed DEA-48, LD authorization memo, DEA-7, and DEA-113 from the original evidence

v. Net weight and description of each prepared PTP sample

vi. Gross weight of each sample and source material PSEE

vii. Signed DEA-12s from each laboratory confirming receipt

viii. A list of the LIMS case numbers from each analyzing laboratory

ix. Summary Report

b. Each analyzing laboratory retains the following:

i. PTP sample number

ii. Copy of the Summary Report

iii. Copy of feedback provided to the forensic chemist (FC).

2. IPTP

a. IPTP sample number

b. LIMS case number of the original analysis

c. A copy of the completed DEA-48 and LD authorization memo

i. For SFL1 REDACTED IPTPs, a LD authorization memo

d. Copy of feedback provided to the FC.

7502.7 External Proficiency Test Samples

The LQAM or designee:

A. Ensures laboratory participation in an EPTP for drug analysis.

EXEMPTION: EPTP requirements do not apply to SFT.

B. Receives feedback from SFQ regarding the EPTP results.

7502.7.1 Completing an EPTP

The LQAM or designee:

A. Obtains one general chemistry sample each fiscal year.

B. Assigns FCs on a rotating basis to analyze the EPTP sample.

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. Does not report DEA-required identifications such as non-controlled substances and quantitative results in the “Comments” section.

F. Forwards the LIMS case number to SFQ.

G. Maintains in LIMS the following information related to each EPTP sample analyzed:

1. EPTP sample number

2. CDR

3. Analytical data

4. Completed vendor’s report

5. DEA-113

6. Copy of feedback provided to the FC.

7502.8 Laboratory Blind Proficiency Test Samples

The SFQAM or designee:

A. Coordinates with IN for the submission of blind proficiency samples.

EXEMPTION: BPTP requirements do not apply to SFT.

7503 VALIDATING AND VERIFYING QUALITATIVE METHODS

Qualitative method validation and verification ensures:

A. Objective evidence is maintained demonstrating that the method is reliable and 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 capable of reliably identifying 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. All methods used for DEA analyses (i.e. enforcement and REDACTED).

I. Implementation of a newly developed or externally published, but non-validated method

J. After modification of method parameters that result in a new method per Exhibit 3/7503

Method verification is required for the following:

K. Transfer of a validated method to other instruments (within or between laboratories)

L. After the disassembly and reassembly of an instrument (e.g. to move to a new laboratory)

M. Implementation of an externally published and validated method (e.g. ASTM test method)

N. In consultation with SFQ and MDTT, method verification testing of applicable performance characteristics to establish a modified method is fit for the intended use may be permitted for previously validated methods that have been modified such that full method validation is not required (e.g. change in ionization source for mass spectrometer).

The LD or designee:

O. Notifies SFQ of any analysis needs not met by existing validated methods.

The FC or designee:

P. Uses verified laboratory reference materials for all method validation or verification testing.

Q. Ensures instrument performance verification is completed per 7506 prior to method validation or verification.

1. For new instrumentation, performance verification and method validation or verification can be performed simultaneously.

R. Validates or verifies:

1. Separation methods per 7503.1 or 7503.3.

a. Methods using soft-ionization mass spectrometry with no fragmentation are validated or verified per 7503.2.1.2.1 or 7503.4.

2. Confirmation methods per 7503.2 or 7503.4.

3. Both the separation and confirmation methods of hyphenated instruments.

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

4. Non-instrumental methods per 7503.6 – 7503.9.

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

T. Submits a validation or verification packet 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

U. Submits selectivity or accuracy data for compounds not previously included in the validation for review.

The QAS or designee:

V. Reviews the validation or verification packet for accuracy and completeness.

W. Ensures the documentation is retained.

X. Submits the reviewed packet to the LQAM.

Y. 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:

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

AA. For method verifications, submits the final reports to the Laboratory Document Control Officer (LDCO).

BB. For method validations, submits the final reports to SFQ.

7503.1 Validating Qualitative Separation Methods

The LQAM or designee:

A. Defines the scope of the method as general-purpose or limited-purpose.

B. Validates qualitative separation methods - GC, LC, or CE - using the following characteristics:

1. Selectivity
2. Repeatability (short-term precision)
3. Reproducibility (long-term precision)
4. Ruggedness (for standardized methods)

7053.1.1 Selectivity

7503.1.1.1 Validation Procedures

The LQAM or designee:

A. For general-purpose methods, prepares solution(s) containing at least:

1. Dimethyl sulfone, methamphetamine, phenyltetrahydroimidazothiazole (PTHIT), cocaine, heroin, oxycodone, fentanyl, and trazodone.
    a. 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, any additional compounds expected to be present in unknown samples (e.g. associated alkaloids, derivatization byproducts, manufacturing byproducts).

B. For limited-purpose methods, 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. Analyzes one injection of the negative control with internal standard and the test solutions.

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 ($S/N_{pk\text{-}pk}$) for each compound.

G. Submits selectivity data for compounds not originally included in the validation to SFQ.

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.

7503.1.1.2 Acceptance Criteria

The LQAM or designee:

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.
   a. When the detector used precludes detection of dimethyl sulfone and an alternative compound is 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 $S/N_{pk\text{-}pk} = 3$ 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.

7503.1.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

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

B. Documents method limitations

7503.1.2 Repeatability (Short-term Precision)

7503.1.2.1 Validation Procedures

The LQAM or designee:

A. For general-purpose methods, prepares solution(s) containing, at minimum, three compounds plus an internal standard. One early-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. One early-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.

7503.1.2.2 Acceptance Criteria

The LQAM or designee:

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.

a. 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.

7503.1.2.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

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

B. Documents method limitations.

7503.1.3 Reproducibility (Long-term Precision)

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

7503.1.3.1 Validation Procedures

The LQAM or designee:

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.

D. Completes and submits for review a final report upon completion of reproducibility testing.

7503.1.3.2 Acceptance Criteria

The LQAM or designee:

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.
    a. During casework analysis, positive controls must be analyzed within the limited validated timeframe.

7503.1.3.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The retention/migration time and relative retention/migration time for each compound tested each week

B. Documents method limitations

7503.1.4 Ruggedness Testing

SFQ:

A. 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 method verification per 7503.3.

C. Submits the final verification packet to SFQ for review.

SFQ:

D. Evaluates the data and prepares a method validation packet consisting of the validation report and the verification reports.

E. Posts the method validation packet to the SFDCC.

7503.2 Validating Qualitative Confirmatory Methods

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

The LQAM or designee:

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

a. Soft-ionization mass spectrometry techniques that do not yield fragmentation are not confirmatory but may be used as separatory techniques and shall be validated per 7503.2.1.2.1.

7503.2.1 Accuracy

7503.2.1.1 Validation Procedures

The LQAM or designee:

A. For general-purpose methods, tests the following:

1. MS: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.

a. When the detector used precludes detection of dimethyl sulfone, the limitation is documented and an alternative compound may be used with SFQ approval.

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. 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.

1. For methods validated at SFL1, structural confirmation of the data is acceptable when no external reference is available.

F. Submits accuracy data for compounds not previously included in the validation to SFQ.

7503.2.1.2 Acceptance Criteria

The LQAM or designee:

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

7503.2.1.2.1 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.

a. When a single compound exhibits variations in base peak, multiple spectra may be evaluated, documented, and deemed acceptable (e.g., due to spectral tilting, MS/MS relative intensities).

2. EI-MS spectra obtained in different carrier gases (i.e. helium and hydrogen) are acceptable for comparison provided acceptance criteria is met.

   a. If acceptance criteria is not met, EI-MS spectra obtained in the same carrier gas must be used for comparison.

3. 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.

4. Collision-induced dissociation (CID) data and in-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.

   3. 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.

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.

7503.2.1.2.2 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.

7503.2.1.2.3 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 (13C-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 and approved by SFQ.

C. No unexplainable extraneous signals are observed in the sample spectrum.

7503.2.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

    1. The accuracy results

    2. The source of the reference spectrum used for spectral comparisons.

        a. For SFL1, when structural confirmation is used in lieu of a reference spectrum, document how the chemical structure results in the observed spectrum.

B. Documents method limitations.

7503.3 Verifying Qualitative Separation Methods

The LQAM or designee:

A. Verifies qualitative separation methods - GC, LC, or CE - using the following characteristics:

    1. Selectivity (selected compounds only)

    2. Repeatability (short-term precision)

    3. Reproducibility (long-term precision)

7503.3.1 Selectivity

7503.3.1.1 Verification Procedures

The LQAM or designee:

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 early eluting compound may be used with SFQ approval.

    2. The same internal standard or fixed compound as the original validation.

3. 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 ($S/N_{pk\text{-}pk}$) for each compound.

7503.3.1.2 Acceptance Criteria

The LQAM or designee:

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

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.

7503.3.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The retention/migration time (t_R, t_m), relative retention/migration time, and $S/N_{pk\text{-}pk}$ for each compound tested.
2. The minimum acceptable retention time where $t_R = 2t_o$.
3. The original validation relative retention/migration times.
4. The relative difference between the relative retention/migration times observed during method verification and those observed during the original validation.

7503.3.2 Repeatability (Short-term Precision)

7503.3.2.1 Verification Procedures

The LQAM or designee:

A. Performs repeatability per 7503.1.2.1 and 7503.1.2.2.

B. Reports the following:

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

C. Documents method limitations.

7503.3.3 Reproducibility (Long-term Precision)

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

7503.3.3.1 Verification Procedures

The LQAM or designee:

A. Performs reproducibility per 7503.1.3.1 and 7503.1.3.2.

B. Reports the following:

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

C. Documents method limitations

D. Completes the reproducibility section of the report.

7503.4 Verifying Qualitative Confirmatory Methods

The LQAM or designee:

A. Verifies confirmatory methods - MS, IR, Raman, NMR - using the following characteristics:

1. Accuracy (selected compounds only)

a. Soft-ionization mass spectrometry techniques that do not yield fragmentation are not confirmatory but may be used as separatory techniques and verified per 7503.4.

7503.4.1 Accuracy

7503.4.1.1 Verification Procedures

The LQAM or designee:

A. For general-purpose methods, tests the following:

1. MS: Dimethyl sulfone, methamphetamine, PTHIT, cocaine, heroin, oxycodone, fentanyl, and trazodone.

NOTE: When the detector used precludes detection of dimethyl sulfone, include the alternative early eluting compound evaluated during validation.

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.

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.

7503.4.1.2 Acceptance Criteria

The LQAM or designee:

A. Evaluates the data per 7503.2.1.2.

B. Reports the following:

1. Accuracy results

2. The source of the reference spectrum used for spectral comparisons.

7503.5 Validating and Verifying the Cannabis Analysis Separatory Method

7503.5.1 Validation Procedures

The LQAM or designee:

A. Performs and evaluates selectivity per 7503.1.1.

B. Performs and evaluates repeatability per 7503.1.2.

1. Calculates the delta-9-tetrahydrocannabinol (THC):IS ratio for each of the injections and documents the relative standard deviation (RSD) of the 30 injections.

C. Performs and evaluates reproducibility per 7503.1.3.

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.

7503.5.1.1 Reporting Requirements

The LQAM or designee:

A. Documents results using the SFL1 method specific report template.

B. Documents method limitations.

7503.5.2 Verification Procedures

The LQAM or designee:

A. Performs and evaluates selectivity per 7503.1.1.

B. Performs and evaluates repeatability per 7503.1.2.

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.

7503.5.2.1 Reporting Requirements

The LQAM or designee:

A. Documents results using the THCSCRN Field Labs Verification Report.

B. Documents method limitations.

7503.6 Validating Qualitative Non-Instrumental Methods

The LQAM or designee:

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 previously included in the validation according to 7503.7.

D. Adds interfering and non-interfering compounds (that are not target analytes) not previously included in the validation according to 7503.8.

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

7503.6.1 Selectivity

75033.6.1.1 Validation Procedure

The LQAM or designee:

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, any additional compounds expected to be present in unknown samples (e.g. associated alkaloids, derivatization byproducts, manufacturing 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.

7503.6.1.2 Acceptance Criteria

The LQAM or designee:

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.

7503.6.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:
   1. The result (e.g. precipitate formation)
B. Documents method limitations (e.g. false negative or false positive results)

7503.6.2 Limit of Detection

7503.6.2.1 Validation Procedures

The LQAM or designee:

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.

7503.6.2.2 Acceptance Criteria

The LQAM or designee:

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.

7503.6.2.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The LOD for each target analyte as the lowest amount where all five replicates produce a positive result.

7503.6.3 Repeatability (Short-term Precision)

7503.6.3.1 Validation Procedures

The LQAM or designee:

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.

7503.6.3.2 Acceptance Criteria

The LQAM or designee:

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.

7503.6.3.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The repeatability results.

7503.6.4 Reproducibility (Long-term Precision)

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

7503.6.4.1 Validation Procedures

The LQAM or designee:

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.

E. Completes a final report and submits to the QAS upon completion of reproducibility testing.

7503.6.4.2 Acceptance Criteria

The LQAM or designee:

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.

1. During casework analysis, positive controls must be analyzed within the established validated timeframe.

7503.6.4.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The reproducibility results.

7503.6.5 Ruggedness Testing

SFQ:

A. Assigns the verification of the validated method to a minimum of two other laboratories.

The assigned laboratory:

B. Performs the verification of the method per 7503.9.

C. Submits the final verification packet to SFQ for review.

SFQ:

D. Evaluates the data and prepares a packet consisting of the validation report and the verification reports.

E. Posts the packet to the SFDCC.

7503.7 Adding Additional Target Analytes

NOTE: This section does not apply to additional compounds that are not new target analytes for the method (see 7503.8).

7503.7.1 Selectivity

7503.7.1.1 Validation Procedures

The LQAM or designee:

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.

7503.7.1.2 Acceptance Criteria

The LQAM or designee:

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.

7503.7.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

   1. The result (e.g. precipitate formation)

B. Documents method limitations (e.g. false negative and false positive result)

7503.7.2 Limit of Detection

7503.7.2.1 Validation Procedures

The LQAM or designee:

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.

7503.7.2.2 Acceptance Criteria

The LQAM or designee:

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

7503.7.2.3 Reporting Requirements

The LQAM or designee:

A. Documents the following:
    1. The LOD for each target analyte as the lowest amount where all five replicates are positive

7503.8 Adding Additional Non-Target Analyte Compounds

7503.8.1 Selectivity

7503.8.1.1 Validation Procedures

The LQAM or designee:

A. Tests a negative control in triplicate.

B. Tests the additional compound in triplicate.

7503.8.1.2 Reporting Requirements

The LQAM or designee:

A. Submits the selectivity results to SFQ.

7503.9 Verifying Qualitative Non-Instrumental methods

A. Verification of color, precipitate, and microcrystalline test methods is accomplished by testing the reagent reliability per 7512 or testing both contemporaneous positive and negative controls.

B. Verification of immunoassay methods is accomplished by testing both contemporaneous positive and negative controls.

7504 VALIDATING AND VERIFYING QUANTITATIVE METHODS

Quantitative method validation and verification ensures:

A. Objective evidence is maintained demonstrating that the method is reliable and performs as intended.

B. Analysts are aware of known method limitations.

C. Methods are capable of reliably determining the purity of the target analyte included within the validation scope.

A quantitative method includes:

D. The technique

E. Sample preparation procedures

F. Operating parameters

Method validation is required for the following:

G. All methods used for DEA analyses (i.e. enforcement and REDACTED)

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 Exhibit 3/7503

Method verification is required for the following:

J. Transfer of a validated method to other instruments (within or between laboratories)

K. After the disassembly and reassembly of an instrument (e.g. to move to a new laboratory)

L. Implementation of an external published and validated method (e.g. ASTM)

M. In consultation with SFQ and MDTT, method verification testing of applicable performance characteristics to establish a modified method is fit for the intended use may be permitted for previously validated methods that have been modified such that full method validation is not required.

The LD or designee:

N. Notifies SFQ of any analysis needs not met by existing validated methods.

The FC or designee:

O. Uses verified laboratory reference materials or previously authenticated materials for all method validation or verification testing.

1. Use a certified reference material (CRM) for target analyte(s).

P. Uses traceable, calibrated equipment to prepare linearity, LOQ, repeatability, and calibrant solutions.

Q. Applies minimum net weight requirements during preparation of all solutions.

R. Ensures instrument performance verification is completed per 7506 prior to method validation or verification.

1. For new instrumentation, performance verification and method validation or verification can be performed simultaneously.

S. Validates or verifies methods per 7504.1 or 7504.3.

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

U. Submits a validation or verification packet 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

V. Submits selectivity data for compounds not previously included in the validation for review provided the method scope permits.

The QAS or designee:

W. Reviews the validation or verification packet for accuracy and completeness.

X. Ensures the documentation is retained.

Y. Submits the reviewed packet to the LQAM.

Z. For compounds not previously included in the validation:

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

OR

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

The LQAM or designee:

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

BB. For method verifications, submits the final reports to the LDCO.

CC. For method validations, submits the final reports to SFQ.

7504.1 Validating Quantitative Methods

The LQAM or designee:

A. Validates quantitative methods using the following performance characteristics:

1. Selectivity
2. Linearity and Limit of Quantitation (LOQ)
3. Repeatability
4. Accuracy
5. Ruggedness

B. For methods that permit multiple sample preparations (e.g. base extraction), validates quantitative methods using the above performance characteristics for each sample preparation, with the exception of selectivity.

7504.1.1 Selectivity

7504.1.1.1 Validation Procedures

7504.1.1.1.1 Separatory Methods

The LQAM or designee:

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 substances routinely found in exhibits containing 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

7504.1.1.1.2 Ultraviolet/Visible Methods

The LQAM or designee:

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. Other substances routinely found in exhibits containing the target analyte.
3. Two-component mixtures containing the target analyte and each selected compound.
    a. Keep the concentration of the target analyte constant for all tests.

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

7504.1.1.2 Acceptance Criteria

7504.1.1.2.1 Separatory Methods

The LQAM or designee:

A. Evaluates the data using the following criteria:

1. The target analyte and internal standard have a single peak with a clear, non-splitting apex.
2. Peak fronting or tailing of the critical pair compounds does not interfere with the resolution from the target analyte or internal standard.
3. The target analyte and internal standard have a retention time >$2t_o$.
4. The target analyte and internal standard, are resolved ($R \geq 1.5$) from each compound tested.

7504.1.1.2.2 UV/Vis Methods

The LQAM or designee:

A. Evaluates the UV/Vis data to ensure compounds have an absorbance <2% relative to the absorbance of the target analyte.

7504.1.1.3 Reporting Requirements

7504.1.1.3.1 Separatory Methods

The LQAM or designee:

A. Reports the following for each compound tested:
    1. Retention time (t_R)
    2. Relative retention time (RRT) relative to the target analyte
    3. Relative retention time (RRT) relative to the internal standard
    4. Resolution from the target analyte
    5. Resolution from the internal standard
B. The minimum acceptable retention time where $t_R = 2t_o$.
C. Documents method limitations.

7504.1.1.3.2 UV/Vis Methods

The LQAM or designee:

A. Reports the following for each compound and mixture tested:
    1. Concentration
    2. Absorbance at the wavelength selected for quantitation analysis.
B. Documents method limitations.

7504.1.2 Linearity and Limit of Quantitation

7504.1.2.1 Validation Procedures

The LQAM or designee:

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

7504.1.2.2 Data Evaluation

The LQAM or designee:

A. Populates the linearity and LOQ worksheet on the appropriate blank form on the SFDCC.
B. Plots the average instrument response or average instrument response ratio (for methods using an IS), y, as a function of concentration, x.
C. Performs a linear regression analysis using all tested concentrations, excluding 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. Determine the overall average sensitivity across the concentration range tested.
3. Determine the sensitivity limits by multiplying the overall average sensitivity by 95% and 105%.
4. Plot the average sensitivity per concentration, the overall average sensitivity, the 95% limit, and the 105% limit as a function of concentration using a logarithmic scale.

7504.1.2.3 Limit of Quantitation

The LQAM or designee:

A. Using the linearity data accepted in 7504.1.2.2, determines the standard deviation of y-intercept (σ) and m (slope of the linearity function).

B. Determines the LOQ using the following equation:

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

7504.1.2.4 Acceptance Criteria

The LQAM or designee:

A. Accepts linearity data for concentrations that fall within the 95 - 105% sensitivity limits.

1. Remove any data points that do not meet the acceptance criteria and regenerate the linear regression analysis and linearity and sensitivity plots.

B. Ensures the final accepted linear range is determined using at least seven concentrations.

7504.1.2.5 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. Each tested solution concentration
2. Average instrument response or instrument response ratio
3. Average sensitivity result
4. Linear regression plot(s) with the slope (m), y-intercept (b), and the correlation coefficient (r)
5. Sensitivity plot(s)
6. The established linear range of the method and the LOQ results.

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

7504.1.3 Repeatability (Short-Term Precision)

7504.1.3.1 Validation Procedures

The LQAM or designee:

A. Evaluates repeatability by analyzing at least two solutions, representing the low and high ends of the linear range.

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

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 RSD of the five analyses at each concentration level tested.

E. Calculates the RSD from the sample standard deviation as follows:

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

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

where X_i is the value obtained from the instrument response or instrument response ratio, n is the number of determinations and $\overline{X}$ is the mean of the values obtained.

7504.1.3.2 Acceptance Criteria

The LQAM or designee:

A. Accepts repeatability data that are within 2% RSD.

7504.1.3.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The calculated RSD for each concentration level.
2. The established repeatability range of the method.
    a. The repeatability range of the method is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.

7504.1.4 Accuracy

7504.1.4.1 Validation Procedures

The LQAM or designee:

A. Evaluates the method accuracy and recovery of the target analyte within the scope of the method to include:

1. The range of anticipated target analyte purity levels (e.g. 5%, 50%, 95% w/w).
2. Target analyte concentrations spanning the entire linear range

3. Mixtures representing each sample matrix (e.g. powder, liquid, tablets)
    a. Powder or tablet sample matrices may be simulated by including commonly encountered adulterants, diluents, alkaloids, and synthetic byproducts in accuracy solutions.
        i. Authentic tablets/capsules, should be used, if available.
    b. Liquid sample matrices may be simulated by preparing accuracy solutions in an appropriate solvent or other liquid.
4. Each target analyte salt forms

B. Prepares at least nine accuracy solutions to evaluate the scope of the method.
1. At each of three purity levels (low, middle, and high purity e.g., 5%, 50%, and 95% w/w), prepare at least three solutions with a target analyte concentration representing the low, middle, and high end of the established linear range.
    a. Factor the documented purity of the CRM into the final solution concentrations and purity values.
    b. Each sample matrix included in the scope of the method must be tested with at least one purity level and three target analyte concentrations. These solutions may be included in the nine accuracy solutions.
        i. When testing additional sample matrices, test solutions at concentrations outside the original accuracy testing solutions to ensure the widest possible working range.
    c. Each salt form included in the scope of the method must be tested with at least one purity level and three target analyte concentrations. These solutions are in addition to the nine accuracy solutions for the first salt form.
        i. Test solutions at concentrations outside the original accuracy testing solutions to ensure the widest possible working range.
2. The following table offers examples of nine accuracy test solutions for an accepted linear range of 0.10 – 1.50 mg/mL of a single salt form. This example would validate the method for tablets and powders, provided acceptable accuracy data is obtained. This example would not validate the method for liquid samples.

Solution	Purity (% w/w)	Target Analyte Concentration (mg/mL)	Sample Matrix
1	5	0.10	Tablet Mimic
2		0.75	
3		1.50	
4	50	0.10	Powder
5		0.75	
6		1.50	
7	95	0.10	Powder
8		0.75	
9		1.50	

C. Quantitates each of the nine solutions using the specified method, and according to 7526.

7504.1.4.2 Acceptance Criteria

The LQAM or designee:

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

$$\%\ error = \frac{(Measured\ purity - Known\ purity)}{Known\ purity} \times 100$$

7504.1.4.3 Reporting Requirements

The LQAM or designee:

A. Reports the following for each solution tested:
    1. Sample preparation
    2. Known purity, including any correction for RM purity or salt form conversion
    3. Experimentally measured purity
    4. Percent difference from the known purity value.
B. Reports the following:
    1. The established accuracy range of the method.
        a. The accuracy range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.
    2. The sample matrices and salt forms that fulfill the acceptance criteria.

7504.1.5 Working Range

The LQAM or designee:

A. Determines and documents the working range of the method.
    1. The working range of a method represents the concentrations tested which fulfill all acceptance requirements for linearity, LOQ, repeatability, and accuracy.
    2. For additional salt forms and matrices, limit the working range to the narrowest range of concentrations that meet the accuracy acceptance criteria.

7504.1.6 Ruggedness Testing

SFQ:

A. 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 method verification per 7504.3.
C. Submits the final verification packet to SFQ for review.

SFQ:

D. Evaluates the data and prepares a method validation packet consisting of the validation report and the verification reports.

E. Posts the method validation packet to the SFDCC.

7504.2 Adding to Validations

7504.2.1 Adding to Selectivity

The LQAM or designee:

A. Performs additional validation testing for the following:

1. The appearance of a component not previously included in the method validation.

a. Minimum Required Performance Characteristics:

i. Selectivity

b. Submits selectivity data to SFQ.

SFQ:

B. Updates the master selectivity worksheet in the Quantitative Validation Spreadsheet.

C. Updates validation documentation to list any additional limitations.

7504.2.2 Adding New Target Analyte Salt Forms or Sample Matrices

The LQAM or designee:

A. Notifies SFQ of any analysis needs not met by existing validated methods such as the appearance of a new target analyte salt form, pharmaceutical preparation, or sample matrix (to include new release formulations or different compositions).

SFQ:

B. Assigns additional validation testing.

The assigned laboratory:

C. Performs additional validation testing.

1. Minimum Required Performance Characteristics:

a. Selectivity (if additional components are present that were not included in the original selectivity testing)

b. Accuracy

D. Prepares an appendix to the validation report.

E. Submits the appendix and data to SFQ.

SFQ:

F. Updates the master accuracy worksheet in the Quantitative Validation Spreadsheet.

G. Updates validation documentation to list any additional limitations.

H. Posts the appendix to the validation report on the SFDCC.

7504.3 Verifying Quantitative Methods

The LQAM or designee:

A. Verifies quantitative methods using the following minimum performance characteristics:
   1. Selectivity
      a. Separation methods: closest-eluting critical resolution pairs
   2. Linearity and LOQ
   3. Quality Control (QC) Check
B. For methods that permit multiple sample preparations (e.g. base extraction), verifies quantitative methods using the above performance characteristics for each sample preparation, with the exception of selectivity.

7504.3.1 Selectivity

7504.3.1.1 Verification Procedures

7504.3.1.1.1 Separatory Methods

The LQAM or designee:

A. Individually analyzes a solution(s) containing the selected critical resolution pair(s):
   1. The target analyte and its closest eluting critical resolution compound
   2. The internal standard (if used) and its closest eluting critical resolution compound.
B. 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.

7504.3.1.2 Acceptance Criteria

7504.3.1.1.1 Separatory Methods

The LQAM or designee:

A. Evaluates the data using the following criteria:
   1. The target analyte and internal standard have a single peak with a clear, non-splitting apex.
   2. Peak fronting or tailing of the critical pair compounds does not interfere with the resolution from the target analyte or internal standard.
   3. The target analyte and internal standard, are resolved ($R \geq 1.5$) from each compound tested.
   4. The target analyte and internal standard have a retention time > $2t_o$.

7504.3.1.3 Reporting Requirements

7504.3.1.3.1 Separatory Methods

The LQAM or designee:

A. Reports the following for each compound tested:
    1. Retention time (t_R)
    2. Relative retention time (RRT) relative to the target analyte
    3. Relative retention time (RRT) relative to the internal standard
    4. Resolution from the target analyte
    5. Resolution from the internal standard
B. The minimum acceptable retention time where $t_R = 2t_o$.

7504.3.2 Linearity and Limit of Quantitation

7504.3.2.1 Verification Procedures

The LQAM or designee:

A. Performs and reports linearity and LOQ per 7504.1.2

7504.3.3 Quality Control Check

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

7504.3.3.1 Verification Procedures

The LQAM or designee:

A. Prepares two QC solutions at concentrations representing the low and high ends of the working range.
    1. For method verification, QC concentrations may be outside the working range provided the measured purity values meet the acceptance criteria.
B. For separatory methods, quantitates each QC solution three times using the test method.
C. For UV/Vis methods, quantitates each QC solution five times using the test method.
D. Measures the instrument response or instrument response ratio for each analysis.
E. Calculates the RSD of the replicate analyses of each QC solution.
F. Calculates the RSD from the sample standard deviation as follows:

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

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

where X_i is the value obtained from the instrument response or instrument response ratio, n is the number of determinations and $\overline{X}$ is the mean of the values obtained.

7504.3.3.2 Acceptance Criteria

The LQAM or designee:

A. Accepts QC check data that are within 2% RSD.

B. Accepts QC check data where the experimentally measured average purity is within ± 5% (relative) from the known purity.

7504.3.3.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:

1. The calculated RSD for each QC solution.
2. The known purity
3. The experimentally measured average purity
4. The % difference from the known purity value for each QC solution.

7504.3.4 Working Range

The LQAM or designee:

A. Determines the working range of the method.

1. The working range of a method cannot exceed the working range established during method validation.
2. The working range is further narrowed by the concentrations tested which fulfill all acceptance requirements for linearity and LOQ during method verification.

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

7505 VALIDATING AND VERIFYING QUANTITATIVE NMR METHODS

Quantitative method validation and verification ensures:

A. Objective evidence is maintained demonstrating that the method is reliable and performs as intended.

B. Analysts are aware of known method limitations.

C. Methods are capable of reliably determining the purity of the target analyte included within the validation scope.

A quantitative method includes:

D. The technique

E. Sample preparation procedures

F. Operating parameters

Method validation is required for the following:

G. All methods used for DEA analysis (i.e. enforcement and REDACTED)

1. The Reference Material Program (RMP) at SFL1 follows the RMP specific SOP

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 Exhibit 3/7503

J. After replacement of the instrument probe

1. Only instrument specific tests in 7505.1.A.1 are required.

Method verification is required for the following:

K. Transfer of an existing validated method to other instruments (within or between laboratories)

NOTE: Quantitative NMR (qNMR) methods validated on one instrument may be transferred to another instrument if the magnetic field strength of the receiving instrument is equivalent to or higher than the magnetic field strength used during validation.

L. After the disassembly and reassembly of an instrument (e.g. to move to a new laboratory)

M. Implementation of an external published and validated method (e.g. ASTM)

N. After repair of the instrument probe

O. In consultation with SFQ and MDTT, method verification testing of applicable performance characteristics to establish a modified method is fit for the intended use may be permitted for previously validated methods that have been modified such that full method validation is not required.

The LD or designee:

P. Notifies SFQ of any analysis needs not met by existing validated methods.

The FC or designee:

Q. Uses verified laboratory reference materials or previously authenticated materials for all method validation or verification testing.

1. Use a CRM for the target analyte and internal standard (calibrant).

R. Uses traceable, calibrated equipment to prepare linearity, LOQ, repeatability, and calibrant solutions for accuracy testing or QC checks.

S. Applies minimum net weight requirements during preparation of all solutions, including preparation of qNMR internal standard solution (ISS).

T. Ensures instrument performance verification is completed per 7506 prior to method validation or verification.

U. Validates or verifies methods per 7505.1 or 7505.5.

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

W. Submits a validation or verification packet 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

X. Submits selectivity data for compounds not previously included in the validation for review provided the method scope permits.

The QAS or designee:

Y. Reviews the validation or verification packet for accuracy and completeness.

Z. Ensures the documentation is retained.

AA. Submits the reviewed packet to the LQAM.

BB. For compounds not previously included in the validation:

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

   OR

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

The LQAM or designee:

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

DD. For method verifications, submits the final reports to the LDCO.

EE. For method validations, submits the final reports to SFQ.

7505.1 Validating Quantitative NMR Methods

The LQAM or designee:

A. Validates qNMR methods by evaluating the following:

1. Instrument-specific tests and performance characteristics:
   a. 90° Pulse width and spectral width
   b. Quantitative spectral region uniformity
   c. Linearity
   d. LOQ
   e. Repeatability
2. Analyte-specific performance characteristics:
   a. Selectivity
   b. Accuracy
   c. Ruggedness

7505.2 Instrument-Specific Tests and Performance Characteristics

The LQAM or designee:

A. Performs the following instrumental tests once for a specific instrument and probe.

1. 90° Pulse width and spectral width
2. Quantitative spectral region uniformity
3. Linearity
4. LOQ
5. Repeatability

7505.2.1 90° Pulse Width and Spectral Width

7505.2.1.1 Evaluation Procedure

The LQAM or designee:

A. Determines the 90° pulse width, using the manufacturer's recommended calibration procedures.

B. Obtains a full spectrum.

7505.2.1.2 Acceptance Criteria

The LQAM or designee:

A. Accepts pulse widths that are ≤90° and ≤10 µs.

B. Verifies the spectral width covers at least -1 to 11 ppm.

7505.2.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the pulse width value and spectral width range.

7505.2.2 Quantitative Spectral Region Uniformity

7505.2.2.1 Evaluation Procedure

The LQAM or designee:

A. Prepares or uses a commercially available solution containing a compound with one prominent peak (e.g., dimethyl sulfone in chloroform).

B. Conducts the qNMR experiment on the prepared solution by using an automated method or by setting the NMR parameters, as follows:

1. Set the delay (D1) to at least 5 times the spin-lattice relaxation time (5 x T1) of the signal with the highest T1.
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 0-10 ppm region.
4. Acquire the spectrum.

C. 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.

7505.2.2.2 Acceptance Criteria

The LQAM or designee:

A. Ensures the peak height RSD is less than 2% in the range 0-10 ppm.

7505.2.2.3 Reporting Requirements

The LQAM or designee:

A. Plots the peak height of the prominent peak in each spectrum across the ppm range.

B. Reports the %RSD of the peak heights.

7505.2.3 Linearity

7505.2.3.1 Validation Procedures

The LQAM or designee:

A. Performs a linearity study using d-methamphetamine hydrochloride CRM.

NOTE: The linearity can be assessed using one representative compound producing signals across the acquisition window and a variety of multiplicities due to the direct proportionality between the observed proton signal response and the number of nuclei generating the signal.

B. Prepares at least seven linearity solutions at different target analyte concentration levels using D_2O and maleic acid, all containing the same concentration of internal standard.

C. Acquires a spectrum of each linearity solution using the test method.

D. Integrates the six methamphetamine signals (1.2 (d, 3H), 2.7 (s, 3H), 2.9 (dd, 1H), 3.0 (dd, 1H), 3.5 (m, 1H), and 7.3 (m, 5H) ppm) and the internal standard signal (6.4 (s, 2H) ppm) in the spectrum for each of the linearity solutions.

7505.2.3.2 Data Evaluation

For each of the six methamphetamine signals individually, the LQAM or designee:

A. Populates the linearity and LOQ worksheet on the appropriate blank form on the SFDCC.

B. Plots the results (area ratio, y, as a function of concentration, x), where the area ratio is the area of the selected target-analyte signal divided by the area of the internal standard signal.

C. Performs a linear regression analysis using all tested concentrations, excluding 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 sensitivity (area ratio/amount) for each concentration analyzed.

2. Determine the overall average sensitivity across the concentration range tested
3. Determine the sensitivity limits by multiplying the overall average sensitivity by 95% and 105%.
4. Plot the average sensitivity per concentration, the overall average sensitivity, the 95% limit, and the 105% limit as a function of concentration using a logarithmic scale.

7505.2.3.3 Limit of Quantitation

For each of the six methamphetamine signals individually, the LQAM or designee:

A. Using the linearity data accepted in 5.2.3.2, determines the standard deviation of y-intercept (σ) and m (slope of the linearity function).
B. Determines the LOQ using the following equation:

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

7505.2.3.4 Acceptance Criteria

For each of the six methamphetamine signals individually, the LQAM or designee:

A. Accepts linearity data for concentrations that fall within the 95 - 105% sensitivity limits.
    1. Remove any data points that do not meet the acceptance criteria and regenerate the linear regression analysis and linearity and sensitivity plots.
B. Ensures the final accepted linear range, for each signal, is determined using at least seven concentrations.

7505.2.3.5 Reporting Requirements

For each of the six methamphetamine signals individually, the LQAM or designee:

A. Reports the following:
    1. Each tested solution concentration
    2. Average area ratio
    3. Average sensitivity result
    4. Linear regression plot(s) with the slope (m), y-intercept (b), and the correlation coefficient (r).
    5. Sensitivity plots.
    6. The established linear range of the method and the LOQ results.
        a. The linear range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria for all six analyte signals.

7505.2.4 Repeatability (Short-Term Precision)

7505.2.4.1 Validation Procedures

The LQAM or designee:

A. Evaluates repeatability by analyzing at least two solutions, representing the low and high ends of the linear range.

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

B. Acquires five spectra for each solution using the test method.

C. Integrates the six methamphetamine signals and the internal standard signal in the spectrum for each of the solutions.

For each of the six methamphetamine signals individually, the LQAM or designee:

D. Documents the area ratio for each solution.

E. Evaluates the repeatability of the method by calculating the RSD of the area ratio values for the five spectra collected at each concentration level tested.

F. Calculates the RSD from the sample standard deviation as follows:

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

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

where X_i is the area ratio value, n is the number of determinations and $\overline{X}$ is the mean of the area ratio values obtained.

7505.2.4.2 Acceptance Criteria

The LQAM or designee:

A. Accepts repeatability data when the RSD for each of the six methamphetamine signals at a given concentration are within 2%.

7505.2.4.3 Reporting Requirements

For each of the six methamphetamine signals individually, the LQAM or designee:

A. Reports the following

1. The calculated RSD for the area ratio at each concentration level.

B. The established repeatability range of the method.

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

7505.3 Analyte-Specific Tests

The LQAM or designee:

A. Performs the following analyte-specific tests using the established method for each target analyte in each internal standard/solvent combination.

1. Selectivity
2. Accuracy

7505.3.1 Selectivity

75035.3.1.1 Validation Procedures

The LQAM or designee:

A. Prepares and analyzes individual solutions of the following:
    1. Selected solvent(s) containing a 0-ppm reference compound.
    2. Target analyte in the selected solvent(s) containing a 0-ppm reference compound.
    3. Internal standard in the selected solvent(s) containing a 0-ppm reference compound.
B. Other substances routinely found in exhibits containing the target analyte in the selected solvent(s) containing a 0-ppm reference compound.

7505.3.1.2 Acceptance Criteria

The LQAM or designee:

A. Evaluates the data using the following criteria:
    1. The target analyte and internal standard have integratable signals within the spectral window.
    2. The target analyte and internal standard have signals separated by at least 0.2 ppm from signals from each compound tested.

7505.3.1.3 Reporting Requirements

The LQAM or designee:

A. Reports the following:
    1. The 0-ppm reference compound used.
    2. The chemical shift for each signal for all compounds tested.
B. Documents any method limitations.

7505.3.2 Accuracy

7505.3.2.1 Validation Procedures

The LQAM or designee:

A. Evaluates the method accuracy and recovery of the target analyte within the scope of the method to include:
    1. The range of anticipated target analyte purity levels (e.g. 5%, 50%, 95% w/w).
    2. Target analyte concentrations spanning the entire linear range
    3. Mixtures representing each sample matrix (e.g. powder, liquid, tablets)
        a. Powder or tablet sample matrices may be simulated by including commonly encountered adulterants, diluents, alkaloids, and synthetic byproducts in accuracy solutions.
            i. Authentic tablets/capsules, should be used, if available.
        b. Liquid sample matrices may be simulated by preparing accuracy solutions in an appropriate solvent or other liquid.

4. Each target analyte salt forms

B. Prepares at least nine accuracy solutions for each target analyte to evaluate the scope of the method.

1. At each of three purity levels (low, middle, and high purity e.g., 5%, 50%, and 95% w/w), prepare at least three solutions with a target analyte concentration representing the low, middle, and high end of the established linear range.

a. Factor the documented purity of the CRM into the final solution concentrations and purity values.

b. Each sample matrix included in the scope of the method must be tested with at least one purity level and three target analyte concentrations. These solutions may be included in the nine accuracy solutions.

i. When testing additional sample matrices, test solutions at concentrations outside the original accuracy testing solutions to ensure the widest possible working range.

c. Each salt form included in the scope of the method must be tested with at least one purity level and three target analyte concentrations. These solutions are in addition to the nine accuracy solutions for the first salt form.

i. Test solutions at concentrations outside the original accuracy testing solutions to ensure the widest possible working range.

2. The following table offers examples of nine accuracy test solutions for an accepted linear range of 5 – 100 mg/mL of a single salt form. This example would validate the method for tablets and powders, provided acceptable accuracy data is obtained. This example would not validate the method for liquid samples.

Solution	Purity (% w/w)	Target Analyte Concentration (mg/mL)	Sample Matrix
1	5	5	Tablet Mimic
2		50	
3		100	
4	50	5	Powder
5		50	
6		100	
7	95	5	Powder
8		50	
9		100	

C. Quantitates each of the nine solutions using the specified method, and according to 7526.

D. Ensures each signal from the target analyte is integrated and a purity value calculated.

7505.3.2.2 Acceptance Criteria

The LQAM or designee:

A. Accepts the accuracy results when the experimentally measured purity for each individual signal (expressed in % w/w) is within ± 5% (relative) from the known purity.

$$\%\ error = \frac{(Measured\ purity - Known\ purity)}{Known\ purity} \times 100$$

B. Accepts the signal for use as a quantitation signal when the accuracy results for all solutions tested meet the acceptance criteria.

7505.3.2.3 Reporting Requirements

The LQAM or designee:

A. Reports the following for each solution tested:
    1. Sample preparation
    2. Known purity, including any correction for RM purity or salt form conversion
    3. Experimentally measured purity from each target analyte signal
    4. Percent difference from the known purity value for each target analyte signal

B. Reports the following:
    1. The quantitation signals for each target analyte
    2. The established accuracy range of the method for each target analyte.
        a. The accuracy range is established by the lowest and highest concentration solutions that fulfill the acceptance criteria.
    3. The sample matrices and salt forms that fulfill the acceptance criteria.

7505.3.3 Working Range of the Method

The LQAM or designee:

A. Determines and documents the working range for each target analyte of the method.
    1. The working range of a method represents the concentrations tested which fulfill all acceptance requirements for linearity, LOQ, repeatability, and accuracy.
    2. For additional salt forms and matrices, limit the working range to the narrowest range of concentrations that meet the accuracy acceptance criteria.

7505.3.4 Ruggedness

SFQ:

A. 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 used.

The assigned laboratory:

B. Performs method verification per 7505.5.

C. Submits the final verification packet to SFQ for review.

SFQ:

D. Evaluates the data and prepares a method validation packet consisting of the validation report and the verification reports.

E. Posts the method validation packet to the SFDCC.

7505.4 Adding to Validations

7505.4.1 Adding to Selectivity

The LQAM or designee:

A. Performs additional validation testing for the following:

1. The appearance of a component not previously included in the method validation.

a. Minimum Required Performance Characteristics:

i. Selectivity

B. Submits selectivity data to SFQ.

SFQ:

C. Updates the master selectivity worksheet in the Quantitative Validation Spreadsheet.

D. Updates validation documentation to list any additional limitations.

7505.4.2 Adding New Target Analytes, Salt Forms, or Sample Matrices

The LQAM or designee:

A. Notifies SFQ of any analysis needs not met by existing validated methods such as the need to quantitate an additional target analyte or the appearance of a new salt form, pharmaceutical preparation, or sample matrix (to include new release formulations or different compositions).

SFQ:

B. Assigns additional validation testing to a laboratory.

The assigned laboratory:

C. Performs additional validation testing.

1. Minimum Required Performance Characteristics:

a. Selectivity (if additional components are present that were not included in the original selectivity testing)

b. Accuracy

D. Prepares an appendix to the validation report.

E. Submits the appendix and data to SFQ.

SFQ:

F. Updates the master accuracy worksheet in the Quantitative Validation Spreadsheet.

G. Updates validation documentation to list any additional limitations.

H. Posts the appendix to the validation report on the SFDCC.

7505.5 Verifying Quantitative NMR Methods

The LQAM or designee:

A. Verifies quantitative methods by evaluating the following:
   1. Instrument-Specific Tests and Performance Characteristics:
      a. 90° Pulse width and spectral width
      b. Quantitative spectral region uniformity
      c. Linearity
      d. LOQ
      e. Repeatability
   2. Analyte-Specific Performance Characteristics:
      a. QC Check

7505.5.1 Instrument-Specific Tests and Performance Characteristics

The LQAM or designee:

A. Conducts probe-specific tests per 7505.2.

7505.5.2 Analyte-Specific Performance Characteristics

7505.5.2.1 Quality Control Check

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

7505.5.2.1.1 Verification Procedures

For each target analyte, the LQAM or designee:

NOTE: The method will be verified for use only for those target analytes for which verification has been completed.

A. Prepares two QC solutions at concentrations representing the low and high ends of the working range.
B. Quantitates each QC solution three times using the test method.
C. Documents the experimentally measured purity for each quantitation signal for both concentrations tested.
D. Calculates the RSD per 7505.2.4.1.F using the three spectra collected for each quantitation signal for both concentrations tested.

7505.5.2.1.2 Acceptance Criteria

For each target analyte, the LQAM or designee:

A. Accepts QC check data that are within 2% RSD for each quantitation signal.
B. Accepts QC check data where the experimentally measured average purity is within ± 5% (relative) from the known purity.

7505.5.2.1.3 Reporting Requirements

For each target analyte, the LQAM or designee:

A. Reports the following:

1. The calculated RSD for each quantitation signal for each QC solution.
2. The known purity
3. The experimentally measured average purity
4. The % difference from the known purity value for each QC solution.

7505.5.3 Working Range

The LQAM or designee:

A. Determines the working range of the method.

1. The working range of a method cannot exceed the working range established during method validation.
2. The working range is further narrowed by the concentrations tested which fulfill all acceptance requirements for linearity and LOQ during method verification.

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

7506 MAINTENANCE AND PERFORMANCE VERIFICATION OF INSTRUMENTS AND EQUIPMENT

7506.1 General Maintenance Procedures

The LQAM or designee:

A. Maintains all components of the equipment (e.g. chromatographic system and detector).

B. Performs necessary maintenance immediately after a problem is identified.

C. Prior to performance verification:

1. Performs a check of cleanliness on all components of the equipment.
2. Performs additional maintenance of instruments and equipment according to 7506.2

7506.2 Instrument and Equipment Maintenance Procedures

The LQAM or designee:

A. Performs the following instrument and equipment specific maintenance:

Instrument	Frequency	Parameter	Procedure
Capillary Electrophoresis	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 necessary.
Gas Chromatograph	Monthly	Septum	Replace septum.
	Monthly	Split liner	Replace the split liner.
	Yearly	Split line	Clean or replace.
	Yearly	Gold seal and syringe	Replace the gold seal and syringe.
Mass Spectrometer with Gas Chromatograph	Yearly	Source	Clean the source. Replace if necessary.
	Every 6 months	Pump	Check pump oil if applicable. Replace and/or fill as necessary.
LCMS	As needed	Source	Clean all source parts.
	Every 6 months	Pump	Check pump oil if applicable. Replace and/or fill as necessary.
NMR	Weekly	Liquid nitrogen	Fill to capacity.
	According to manufacturer's specifications	Liquid helium	Fill to capacity.
Microscopes	Yearly	Operation	Verify proper operation

7506.3 General Performance Verification Procedures

The LQAM or designee:

A. Adopts and implements the specific procedures listed below for instruments such as NMR and IR.

B. Adopts standardized performance verification procedures where established.

C. For all other instrumentation, establishes specific performance verification procedures and acceptance criteria that meet the general system requirements listed below and are consistent with the methods used.

D. Reviews and approves these procedures before implementation.

E. 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 or verification and before an instrument can be used for case work.

2. In the sections below, substantial instrument maintenance refers to any non-routine replacement or repair of non-consumable parts and any maintenance performed by non-DEA personnel. (See Exhibit 3/7503).

F. Uses verified reference materials or traceable standards for all performance samples.

G. For separatory instruments, performs the instrument performance verification of the detector (i.e. diode array detector, mass spectrometer, infrared detector) where applicable, prior to evaluation of the separatory component.

H. For equipment that requires calibration by external providers, uses equipment with a current calibration certificate.

I. Labels the following equipment to allow users to readily identify the calibration status:

1. Balances
2. NIST-traceable weights
3. Automatic pipettes
4. Traceable glassware

7506.3.1 Performance Verification Templates

The LQAM or designee:

A. Records specific performance verification procedures.

B. For manufacturer-recommended procedures, notes the reference citation.

C. Maintains a copy of the approved performance verification procedures in each instrument logbook.

7506.4 Instrument Performance Verification Procedures and Acceptance Criteria

7506.4.1 Balances

7506.4.1.1 Environment

The LQAM or designee:

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.

7506.4.1.2 Calibration Check

A. Frequency:

1. Monthly
2. After substantial maintenance
3. After a balance is physically relocated
4. Before a balance is returned or put into service following an external calibration

B. Performance Sample: NIST-traceable, calibrated weights

C. Procedure:

The LQAM or designee:

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 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 when using more than one weight) 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 of each mass.
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 and dual-range balances, considers the balance as two independent balances
    a. Checks the repeatability of the fine-readability range with both high-mass and low-mass reference weights.
    b. Checks the repeatability of the coarse-readability 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 is ≤ 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.

Readability:	Acceptance Range:
0.000001 g	± 0.000008 g
0.00001 g	± 0.00017 g
0.0001 g	± 0.0003 g
0.001 g	± 0.003 g
0.01 g	± 0.04 g
0.1 g	± 0.2 g

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

The LQAM or designee:

1. Repeats the reference weight measurements following the procedures in 7506.4.1.2.
2. Places balance out-of-service.
3. Contacts service provider if problem is not resolved.
4. Returns balance to service when the measurements obtained meet the acceptance criteria.

7506.4.1.3 Calibration

The LQAM or designee:

A. Ensures the balances are calibrated annually by an ISO/IEC 17025-accredited calibration laboratory.

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.

7506.4.1.4 Reference Weights

The LQAM or designee:

A. Utilizes appropriate NIST-traceable calibrated weights that are selected based on balance precision and readability. The following are acceptable weight classes based on balance readability:

Balance Readability	Acceptable Weight Class
0.00001 g – 0.000001 g	UltraClass
	OIML R 111 Class E1
0.0001 g – 0.00001 g	OIML R 111 Class E2
	ANSI/ASTM E617 Class 1
0.01 g – 0.001 g	OIML R 111 Class F1
	ANSI/ASTM E617 Class 2
0.1 g – 0.01 g	ANSI/ASTM E617 Class 3

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

B. 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.

C. Ensures the weights are not altered in any way (e.g. adding distinguishing marks, mishandling that could cause scratching or chipping).

7506.4.1.5 Reference Weight Calibration

The LQAM or designee:

A. Ensures the NIST-traceable weights are calibrated once annually by an ISO/IEC 17025-accredited calibration laboratory.

B. Maintains the calibration certificates.

7506.4.2 Capillary Electrophoresis System

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.2.1 Diode Array Detector

A. Performance Sample: Not applicable

B. Procedure: Perform detector tests and verifications, as recommended by the manufacturer.

C. Acceptance Criteria: Test and verification results are within manufacturer's specifications.

7506.4.2.2.2 Electrophoresis

A. Method: A commonly used method. A general-purpose method must be selected when validated on the instrument.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.

   a. 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).

   a. 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.

   a. 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.

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

D. 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 $S/N_{pk\text{-}pk} = 3$ is observed for each compound tested.

7506.4.3 Gas Chromatography System

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.3.1 Chromatography

A. Method: A commonly used method on each column. A general-purpose method must be selected when validated on the instrument.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.

   a. 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).
    a. 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.
    a. 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.

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

D. 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. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, the t_R of the first eluting compound must be within 0.1 minutes of the t_R obtained during the previous month.
        i. 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 Exhibit 3/7503)
5. A minimum $S/N_{pk\text{-}pk} = 3$ 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 ± 5% relative to the known prepared purity of the QC sample.

7506.4.4 Gas Chromatography-Mass Spectrometry System

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.4.1 Mass Spectrometer Calibration Check

A. Performance Sample: PFTBA

B. Procedure: Perform a standard spectra tune (stune).

C. Acceptance Criteria: Tune results are within the system-wide standardized specifications.

7506.4.4.2 Gas Chromatography

A. Method: A commonly used method on each column. A general-purpose method must be selected when validated on the instrument.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.
    a. 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).
    a. 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.
    a. 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.

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

D. 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. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, the t_R of the first eluting compound must be within 0.1 minutes of the t_R obtained during the previous month.
        i. 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 Exhibit 3/7503)
5. A minimum $S/N_{pk\text{-}pk} = 3$ is observed for each compound tested.

7506.4.5 Gas Chromatography - Infrared Spectrophotometer

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.5.1 IR Detector

A. Performance Sample: A solution containing a semi-volatile substance with a vapor-phase IR spectrum documented in literature.

B. 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.

C. Acceptance Criteria: Measured peak positions of three high intensity absorption bands are within the experimental resolution of the cited reference values.

7506.4.5.2 Gas Chromatography

A. 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.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.

    a. 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).

    a. 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.

    a. 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.

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

D. 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. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, the t_R of the first eluting compound must be within 0.1 minutes of the t_R obtained during the previous month.

        i. 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 Exhibit 3/7503)

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

7506.4.6 Infrared Spectrophotometer

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

1. Perform the transmission check before the reflectance check to verify performance of the instrument prior to verification of performance of the accessory (e.g. attenuated total reflectance (ATR)).

7506.4.6.1 Transmission Wavelength and Resolution Check

A. Method: 8 scans, 4 cm-1 resolution

B. Performance Sample: Traceable Polystyrene

C. Procedure:

1. Collect polystyrene transmittance spectrum

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.

7506.4.6.2 Reflectance Wavelength and Resolution Check (for ATR) of the Accessory

A. Method: IR01

B. Performance Sample: Caffeine

C. Procedure:

1. Collect caffeine spectrum

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.

7506.4.7 Liquid Chromatography System

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.7.1 Diode Array Detector

A. Performance Sample: Not applicable

B. Procedure: Perform detector tests and verifications, as recommended by the manufacturer.

C. Acceptance Criteria: Test and verification results are within manufacturer's specifications.

7506.4.7.2 Chromatography

A. Method: A commonly used method on each column. A general-purpose method must be selected when validated on the instrument.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.
    a. 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).
    a. 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.
    a. 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.

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

D. 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. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, 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 $S/N_{pk\text{-}pk} = 3$ is observed for each compound tested.

E. For quantitation methods, criteria listed above, with the exception of the first eluting compound criteria, and QC solutions are within ± 5% relative to the known prepared purity of the QC sample.

7506.4.8 Liquid Chromatography–Mass Spectrometry System

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument
1. For mass spectrometer calibration, at least every 6 months, after substantial maintenance, and after physically relocating the instrument
2. Prior to use after conversion from another source, perform 7506.4.8.4.

7506.4.8.1 Mass Spectrometer Tune

A. Performance Sample: Laboratory-customized or manufacturer-recommended solution

B. Procedure: Tune the mass analyzer following manufacturer's instructions.

C. Acceptance Criteria: Tune results are within manufacturer's specifications.

EXEMPTION: Triple-quadrupole mass analyzers do not require tunes.

7506.4.8.2 Mass Spectrometer Calibration

A. Performance Sample: Manufacturer-recommended solution

B. Procedure: Calibrate the mass analyzer following the manufacturer's instructions.

C. Acceptance Criteria: Calibration results are within the manufacturer's specifications.

7506.4.8.3 Liquid Chromatography

A. Method: A commonly used method on each column. A general-purpose method must be selected when validated on the instrument.

B. Performance Sample:

1. General-purpose: Mixture containing a minimum of four commonly encountered compounds at known concentrations.
    a. 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).
    a. 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.
    a. 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.

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

D. 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. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, 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 $S/N_{pk\text{-}pk} = 3$ is observed for each compound tested.

7506.4.8.4 Mass Spectrometer Source: Direct Analysis in Real Time (DART), Electrospray Ionization (ESI)

A. Method: A commonly used method.

B. Performance Sample:

1. With liquid chromatography, see 7506.4.8.3.B

2. Without liquid chromatography (i.e. DART, direct infusion), mixture containing a minimum of three commonly encountered compounds at known concentrations.

a. Sample will contain a low, mid, and high mass compound spanning the range of the method and, if included in the scope of the method, one 0.5% low-level marker.

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

D. Acceptance Criteria:

1. The pseudo-molecular ion, to include salt or solvent adduct signals, corresponds to the theoretical molecular weight of the compound.

2. The overall sample fragmentation pattern (relative ion abundances, m/z values, and isotopic distributions) corresponds to the spectrum collected during method validation.

a. Ensure the spectra have the same base peak.

3. The measured m/z values for prominent ions are of the same nominal mass as those in the spectrum collected during method validation.

4. The molecular ion (or pseudo-molecular ion) is observed in the fragmentation spectrum if it is observed in the spectrum collected during method validation.

5. No prominent unexplainable extraneous ions are observed in the fragmentation spectrum.

7506.4.9 Nuclear Magnetic Resonance Spectrometer

7506.4.9.1 High Field (superconducting magnet, typically > 200 MHz)

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

1. For nuclei other than ^{1}H and ^{13}C, (i.e. ^{19}F or ^{15}N), either a sensitivity or a pulse width test must pass manufacturer's specifications prior to acquiring spectra using these nuclei.

7506.4.9.1.1 Proton (^{1}H) Line Shape

A. Performance Sample: Manufacturer's recommended sample

B. Procedure: Measure the non-spinning line shape values using a manufacturer's recommended test

C. 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.

7506.4.9.1.2 Proton (^{1}H) Pulse Width

A. Performance Sample: Manufacturer's recommended sample

B. Procedure: Measure the pulse width using a manufacturer's recommended test

C. Acceptance Criteria: The 90º pulse width is less than or equal to the manufacturer's specification

7506.4.9.1.3 Proton (^{1}H) Sensitivity

A. Performance Sample: Manufacturer's recommended sample

B. Procedure: Measure the signal to noise using a manufacturer's recommended test

C. Acceptance: The signal to noise is greater than or equal to the manufacturer's specification

7506.4.9.1.4 Carbon (^{13}C) Pulse Width

A. Performance Sample: Manufacture's recommended sample

B. Procedure: Measure the pulse width using a manufacturer's recommended test

C. Acceptance Criteria: 90º pulse width is less than or equal to the manufacturer's specifications

7506.4.9.1.5 Carbon (^{13}C) Sensitivity

A. Performance Sample: Manufacturer's recommended sample

B. Procedure: Measure the signal to noise using a manufacturer's recommended test

C. Acceptance Criteria: The signal to noise is greater than or equal to the manufacturer's specification

EXCEPTION: Not required for instruments using an AutoX probe.

7506.4.9.2 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.

7506.4.10 Ultraviolet/Visible Spectrophotometer

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

7506.4.10.1 Wavelength Accuracy

A. Performance Sample: N/A

B. Procedure: Perform wavelength accuracy function

C. Acceptance Criteria: Wavelength of xenon line is within ± 0.5 nm of 541.9 nm

7506.4.10.2 Performance Check

A. Performance Sample: Low QC and high QC solutions for the method selected.

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

C. Acceptance Criteria: QC solutions are within ± 5% relative to the known prepared purity of the QC sample.

7506.4.11 Polarimeter

A. Frequency: Monthly, after substantial maintenance, and after physically relocating the instrument

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.

7506.4.12 Field Equipment

A. Frequency: Monthly and before use at off-site location

7506.4.12.1 Portable IR or Raman

A. Performance Sample: Manufacturer-recommended compound

B. Procedure: Analyzes the sample following manufacturer's instructions.

C. Acceptance Criteria: Verification tests meet manufacturer's specifications.

7506.4.12.2 Ion Mobility Spectrometer

A. Performance Sample: Manufacturer-recommended compound

B. Procedure: Analyze the sample following manufacturer's instructions.

C. Acceptance Criteria: Verification tests meet manufacturer's specifications.

7506.4.12.3 Portable Mass Spectrometer

7506.4.12.3.1 System Check

A. Performance Sample: Manufacturer-recommended sample

B. Procedure: Analyze the sample following manufacturer's instructions.

C. Acceptance Criteria: Verification tests meet manufacturer's specifications.

7506.4.12.3.2 Mass Spectrometer Core Calibration

A. Performance Sample: N/A

B. Procedure: Calibrate the mass spectrometer core following the manufacturer's instructions.

C. Acceptance Criteria: Calibration results are within the manufacturer's specifications.

7506.5 Documentation Requirements

The LD or designee:

A. Specifies the format of the equipment logbook.

B. Archives the equipment logbook in the laboratory for 75 years.

The LQAM or designee:

C. Maintains records and logbooks for calibration checks, performance verifications, and all other repairs and preventative maintenance.

D. Includes in the logbook, where applicable, 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.

F. Evaluates results of performance verification.

G. If the results do not meet acceptance criteria, investigate the nature and cause of any failure and performs maintenance to address the issue.

H. Documents the completion and all results of the performance verification in the instrument logbook.

1. Include a copy of the performance verification results, all data, including data that does not meet acceptance criteria, and reports generated in the instrument logbook.

I. If maintenance is unable to produce data that meets the acceptance criteria, places the instrument out of service.

1. Clearly label the instrument as out of service.
2. Document the instrument status and reason for placing out of service in the logbook.
3. Update the logbook monthly until the problem has been resolved and the instrument can meet the specified acceptance criteria.

J. Ensures that all instrumental data contains the corresponding DEA identification number, date performed, and the name or initials of the individual performing the verification.

7507 FIELD LABORATORY REFERENCE MATERIALS

The LD:

A. Designates a primary RM monitor who will administer the program.

B. Designates alternate RM monitor(s).

C. Restricts access to significant quantities of RMs.

NOTE 1: RMs consist of controlled substances, non-controlled substances, precursors, and new psychoactive substances.

NOTE 2: RMs may be called by different names such as reference standards or analytical standards.

The RM Monitor(s):

D. Obtains RMs from SFL1, reputable commercial sources in accordance with LOM 7000, or another DEA laboratory.

1. Obtains CRMs with a traceable purity value from an ISO/IEC 17025 or 17034 accredited supplier.

E. Ensures quantitative RMs and qNMR internal standards (IS) are CRMs with a current certificate.

F. Treats new psychoactive substance (NPS) 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 7508.

J. Retains verification results in either electronic or hard copy format.

K. Transfers RMs to another DEA laboratory in accordance with LOM 7400.

7507.1 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 7400)

The RM Monitor(s):

B. Classifies stock RMs as quantitative or qualitative materials.

C. Documents RMs known to degrade, absorb water in their storage environment, or become otherwise unstable.

1. These RMs will not be placed in the working RMs inventory unless proper storage conditions are met (e.g. a desiccator).

7507.2 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.

7507.3 Storing Controlled Substances and Listed Chemical Reference Materials

The RM Monitor(s):

A. Stores stock RMs in accordance with LOM 7400.

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 7400.

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 7509.

7507.4 Documentation

7507.4.1 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. Stock RMs must be returned within the same business day of receipt.

i. The entire stock RM may be provided to a chemist for use without creating a working vial.

b. The RM monitor will record the net weight removed, name of recipient and purpose of transfer in the logbook or electronic record.

c. Removal of more than 2 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

7507.4.2 Working Reference Materials

The RM Monitor(s):

A. Maintains a list of working RMs in a bound logbook or electronic format.

B. Maintains sign-out records for the controlled working RM vials.

1. If using the Controlled Working RM Log, a separate log must be used for each RM number.
2. A sign-out record for non-controlled working RM vials may be maintained. The format of the record may be designated by the laboratory.

C. Documents when a controlled working RM vial is replenished.

D. Monthly, identifies controlled working RMs vials that were not accessed in that time-frame.

E. 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.

F. 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 LIMS or the comments section of the Controlled Working RM Log.

The FC:

G. Weighs the working vial at the time the material is accessed and returned.

H. Uses, at a minimum, a 3-place balance to record vial weights.

I. Maintains possession of the controlled working RM vial.

1. The controlled working RM vial cannot be transferred to another analyst before returning the vial.

J. Returns the controlled working RM vial within the same business day of receipt.

K. 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 LIMS or the comments section of the Controlled Working RM Log.

7508 VERIFYING FIELD LABORATORY REFERENCE MATERIALS

A. Verification is the process by which the identity of a RM is assessed to determine if the material is fit-for-purpose.

B. Verifying RMs will not result in revised purity values.

The LQAM or designee:

C. Verifies all RMs used 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.

7508.1 Verifying Qualitative Reference Materials

The RM Monitor 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.

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. Prior to use in casework, verifies optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected (e.g., methamphetamine).

1. Stock and working RM vials shall be clearly denoted as not to be used for enantiomer determination until verified.

2. Subsequent verifications of optical isomeric form are not required after the initial verification.

D. Analyzes the collected data and determines if the RM meets the acceptance criteria.

1. Acceptance Criteria:
    a. Data obtained is consistent with data from authentication, previous verification, a verified reference database from SFL1, a commercial library, published literature spectra, or at least one other ISO/IEC 17025-accredited laboratory.

       NOTE: For RMs obtained from a reputable commercial source, the vendor supplied data may be used for comparison if no other reference data is available.
    b. Data does not indicate any significant changes in the composition of the RM.
    c. There are no unexpected components.

E. Places RMs that meet the acceptance criteria in the RM inventory.

F. For RMs that do not meet the acceptance criteria, does one of the following:
    1. Remove the RM from the inventory and send to SFL1 for purification and authentication.
    2. Transfer the RM to the destruction coordinator.

7508.2 Verifying Certified Reference Materials

The RM Monitor or designee:

A. Upon initial receipt of a CRM, performs a qualitative verification per 7508.1.
    1. For NMR internal standard CRMs, verifies the IS using one confirmatory test.
    2. The reported purity value may be accepted without any additional testing.

B. Ensures CRMs used for quantitation are not used past the expiration date.

C. Disposes of expired CRM.

7508.3 Verifying QC Samples

The RM Monitor or designee:

A. Verifies the identity of the target analyte by performing a confirmation test.
    1. Ensure the data is consistent with stated target analyte of the QC sample.

B. Performs quantitative verification during the quantitative analysis process. (See 7526)

7508.4 Documentation

The RM Monitor or designee:

A. Creates and maintains files for each RM to include at least the following:
    1. Completed Reference Material Verification Summary Sheet or equivalent
    2. Data used for comparison from authentication, previous verification, or a verified reference database from SFL1, a commercial library, published literature spectra, or at least one other ISO/IEC 17025-accredited laboratory.
    3. Laboratory-generated verification and re-verification data

B. Retains files for a minimum of three years from the date the RM is consumed.

7509 MONITORING STORAGE CONDITIONS

7509.1 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 7800 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, solutions, or RMs, when accessed for use, are stored under proper conditions.

The LQAM or designee:

E. Checks the temperature of refrigerators and freezers used for storing evidence, chemicals, reagents, solutions, and RMs which require storage at a temperature other than room temperature.

F. Uses a calibrated thermometer (appropriate for the required temperature range) or calibrated electronic temperature monitoring system to monitor the refrigerators and freezers, according to the criteria shown below.

   1. Refrigerator: >0°C - 10°C (32°F - 50°F)
   2. Freezer: ≤ 0°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 monthly.

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., RM 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, solutions, and RMs.

7510 MEASUREMENT TRACEABILITY

7510.1 Scope

A. Measurement traceability is required for net weight measurements and purity determinations.

7510.2 Net Weight

The FC:

A. Performs net weight measurements using balances that have been calibrated annually by an ISO/IEC 17025-accredited calibration laboratory.
   1. Each balance is identified with a unique traceable number that is documented in the case file for each weight measurement.

7510.3 Purity

The FC:

A. For calibrant solution and qNMR internal standard solution preparation:
   1. Uses CRMs with a traceable purity value from an ISO/IEC 17025 or 17034 accredited supplier.
   2. Obtains weight measurements on balances that have been calibrated annually by an ISO/IEC 17025-accredited calibration laboratory
   3. Uses volumetric equipment that has been calibrated by an ISO/IEC 17025-accredited calibration laboratory.

7511 USE OF EXTERNALLY PROVIDED ANALYTICAL SUPPLIES AND SERVICES

The LD:

A. Ensures providers (vendors) of analytical supplies and services meet the requirements listed below:

Item(s)	Vendor Requirement(s)	Frequency of Requirement
Consumables		
Certified 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

Item(s)	Vendor Requirement(s)	Frequency of Requirement
Weight Calibration	Accreditation through ISO/IEC 17025	Required annually
Automatic Pipette Calibration	Accreditation through ISO/IEC 17025	Required every 6 months
Thermometer or 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	See LOM 7100
Reagents		See 7512

7512 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.

7512.1 Preparing Reagents

The LQAM or designee:

A. Prepares reagents according to the method validation documentation.

7512.2 Documenting Reagents

7512.2.1 Stock Containers

The LQAM or designee:

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

7512.2.2 Secondary Containers

The LQAM or designee:

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

7512.2.3 Commercial (Purchased) Reagents

The LQAM or designee:

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.

7512.3 Labeling Containers

The LQAM or designee:

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.

7512.4 (Re)Verifying Reagents

The LQAM 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
    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
    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.

7512.5 Disposing of Reagents

The LQAM or designee:

A. Disposes of a reagent as hazardous waste when it meets any of the following criteria: (See LOM 7800)

1. Does not produce expected results during (re)verification
2. Drastically changes in appearance or composition
3. Is no longer needed

7513 PEER REVIEW OF FORENSIC CHEMIST EXAMINATIONS

7513.1 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, administrative, and policy compliance review.

C. FCs will not conduct peer reviews of their own work.

7513.2 Conducting Peer Reviews

The LQAM 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.

7513.3 Reviewing the Results of the Peer Review

The LQAM 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.
        a. Follow non-conformance procedures per LOM 7100 as needed.

The FC:

B. Makes any corrections that are required as a result of the peer review.
C. Creates an amended report (if needed) (see 7530.8).
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.

7513.4 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.

7521 EVIDENCE ANALYSIS

7521.1 Scope

A. Sections 7521 through 7540 contain policy and procedures for the various aspects of evidence analysis.
B. Laboratory management must approve all deviations that do not fulfill these minimum requirements.
    1. Document approvals in the Supervisory Approval test

7522 DETERMINING GROSS WEIGHT AND OPENING EVIDENCE

7522.1 Determining Gross Weight

The FC:

A. Ensures all labels (e.g. LIMS label and evidence label) on the evidence are consistent and agree 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.

D. Reports the gross weight in LIMS and on the DEA-113.

7522.2 Opening Evidence

7522.2.1 Properly Sealed Evidence

The FC:

A. Opens plastic sealed evidence envelopes (PSEE) and manila envelopes by cutting along the edge opposite the submitting personnel'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 submitting personnel'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 7800 for the decontamination of biohazard exhibits.

7522.2.2 Improperly Sealed Evidence

The FC:

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

7522.3 Describing the Evidence

The FC:

A. Compares the physical evidence with the description reported by the submitting Investigating Agency (IA).

B. Describes the evidence as received from the outermost packaging to the innermost contents in the Description of Evidence test (see Exhibit 9/7530).

C. Obtains a witness when the physical evidence differs significantly from the description.

1. Discuss the discrepancy with a SC and contact the submitting personnel, if necessary, in an attempt to resolve any significant differences.
2. 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 exhibit.

7522.4 Handling Interior Packaging

The FC:

A. Marks interior packages with initials, date, and unique identifier. Alternatively, seals the interior package(s) into a substitute container marked with initials, date, and unique identifier.
    1. If friction ridge examination is requested, see 7561.
    2. For bulk exhibits, it is only necessary to mark the threshold portion.

7522.5 Photographing Evidence

The FC:

A. During laboratory analysis, digitally photographs the following:
    1. The entire seizure of bulk evidence

        EXCEPTION: Bulk evidence that will not be separated into bulk and threshold portions does not require photographs of the entire seizure.
    2. Evidence during analysis to document any unusual physical feature(s) prior to processing
        a. This includes photographs of the front and back of tablets when no intact tablets will remain after analysis.
    3. REDACTED
B. During field operations, digitally photographs and/or records the following:
    1. Items seized as evidence
    2. Items from which samples are removed for analysis
    3. Essential area(s) where evidence was obtained
C. Attaches all photographs to the Image finding in the Description of Exhibit and Sampling test.

    EXCEPTION 1: REDACTED

    EXCEPTION 2: Photographs taken for a specific analytical test are attached to the Image finding in the corresponding LIMS test.
D. Includes a self-documenting sign in all photographs which contains the following:
    1. LIMS case number
    2. Sub-exhibit number, if applicable
    3. Date photographs are taken
    4. Location of the seizure (bulk evidence only)
    5. Laboratory
    6. Handwritten initials of photographer

    EXCEPTION: REDACTED

E. Positions a measuring device such as a ruler or yardstick in all photographs.

1. For field operations, if a measuring device is unavailable, an object of known size (e.g. dollar bill) may be used to demonstrate scale.

F. Assembles or stacks the evidence to make a clear, visual display of the individual units

G. Photographs the entire display.

1. If the evidence is in opaque containers, open at least one container to display the drug material.

7522.6 Creating Sub-Exhibits

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. Provides a brief description of the sub-exhibit 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.

7523 DETERMINING NET WEIGHT AND UNCERTAINTY OF MEASUREMENT ESTIMATES

The FC:

A. For all exhibits, determines the net weight and the associated uncertainty.

1. For submissions representing a part of a larger seizure (i.e., exemplars) net weight uncertainty estimates are not required to be calculated or reported.

B. For multi-unit exhibits, determines the unit count.

C. For liquids, determines the volume.

7523.1 Observing Minimum Weight Thresholds

The FC:

A. Ensures that following minimum weight thresholds are observed when performing net weight measurements:

Readability (g):	Minimum Weight (g):
0.1	20.0
0.01	2.50
0.001	0.250
0.0001	0.0300
0.00001	0.01500
0.000001	0.001000

B. Applies minimum weight thresholds to the variable being measured (e.g. each individual net weight measurement, not to the total net weight of the exhibit).

1. Minimum weight thresholds do not apply to tared containers (paper, weighing boats, original or substitute packaging, glassware, etc.).
2. When doing container extrapolation, minimum weight thresholds are applied to the individual container weights.

7523.2 Calculating Net Weight

A. For capsule exhibits, the net weight does not include the capsule shell.

B. For impregnated paper, the net weight is to include the paper.

The FC:

C. Determines the total net weight by one of the following:

1. Direct measurement of the contents of all units.
2. Contents extrapolation:
    a. Individually weigh nine or nine groups of randomly selected units.
    b. For 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 needed for analysis.
    c. Calculate the average net weight per unit.
    d. Obtain the total net weight by multiplying the average weight per unit by the total number of units.
3. Container extrapolation:
    a. Determine the total gross weight of the units by direct measurement of all units.
    b. Individually weigh nine or nine groups of randomly selected empty containers.
    c. For 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 needed for analysis.
    d. For exemplar samples submitted in similar packaging and no uncertainty will be reported, weigh one empty container.
    e. Calculate the average weight per empty container.
    f. Obtain the total weight of the empty containers by multiplying the average weight per container by the total number of units in the exhibit.
    g. Obtain the total net weight by subtracting the total weight of the empty containers from the total gross weight for all units.
4. A combination of direct weighing and extrapolation.

7523.2.1 Determining Unit Count

The FC:

A. Determines the total unit count by one of the following:
   1. Counts all units directly.
   2. By extrapolation
      a. Individually weigh nine or nine equal groups of randomly selected dosage units.
      b. Calculate the average weight per dosage unit.
      c. Obtain the total number of dosage units in the exhibit by dividing the total net weight by the average weight per dosage unit.

7523.2.2 Determining Volume

The FC:

A. 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.
B. Calculates the total net volume using the total net weight and the composite's density.

7523.2.3 Internal Body Carry Exhibits

The FC:

A. Determines the total net weight of the exhibit by one of the following:
   1. Direct measurement of the contents of all units.
   2. Contents extrapolation.
      a. Less than nine units may be weighed to limit exposure.

7523.3 Uncertainty of Measurement Estimate Determination for Net Weight

The FC:

A. Reviews the Uncertainty Calculator and applicable Worksheet or Legacy Calculator in LIMS to ensure the values from each weighing event are entered correctly.
B. Accepts the calculated uncertainty when:
   1. For extrapolation cases, the RSD obtained from the 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.

7523.4 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 (Exhibit 4/7523).

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 the following statement on all exhibits:

"The net weight represents the weight of all material, excluding the packaging."

E. Includes a statement, selected from Exhibit 6/7530, that describes the procedure used for net weight determination and the associated uncertainty, if determined, in the *Observations, Results, and Conclusions* section of the DEA-113.

1. 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.

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

1. For extrapolated solid dosage unit counts, include the statement:

"Total number of units is an extrapolated value."

2. Measurement uncertainty values associated with these quantities are not reported on the DEA-113.

7523.5 Revising UMEs

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.

7524 THE EVIDENCE SAMPLING PLAN

The Evidence Sampling Plan (ESP) for Qualitative Analysis:

A. Requires selection of all units for submissions having fewer than 10 units.

B. Requires a statistical sampling approach for submissions consisting of 10 or more units (non-exemplar). This approach:

1. Requires random sampling. (See Exhibit 5/7524)

2. Allows for a population inference at a 95% level of confidence based on one of two approaches:

a. Hypothesis testing using the probability theory of the hypergeometric distribution

b. Generating a confidence interval for the population proportion.

3. Requires additional sampling when no consistent controlled substance or NPS is identified in all units tested.

C. Includes procedures for sampling residue exhibits.

D. Includes procedures for arbitrary sampling when no inference will be made on the population.

E. REDACTED

1. REDACTED

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 Exhibit 5/7524)
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).

7524.1 Sampling for Qualitative Testing

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

7524.1.1 Single and Multi-unit Exhibits

The FC:

A. Separates the exhibit into sub-exhibits based on potentially different populations, as needed.

1. Different populations may be based on different colors, markings, expected target analyte(s), bi-layer liquids, the results of chemical testing, etc.

B. Samples units based on the total number of units in the exhibit.

1. **1-9** Units
   a. Selects all units.
   b. For single unit exhibits when quantitation is anticipated to be required per 7526, proceeds directly to composite formation according to Exhibit 7/7524.
   c. For tablet exhibits, photographs the front and back of an intact unit prior to analysis.
2. 10 or More Units
   a. Randomly selects nine units.
   b. If the exhibit includes multiple containers, samples from as many containers as possible.
   c. Segregates or labels (e.g., numbers) each unit selected.

C. Analyzes each selected unit per 7525.

D. Evaluates results per 7524.2.

E. Forms a composite when directed per 7524.3.

F. For any deviations from the ESP, documents the reason and obtains approval in advance in the Description of Exhibit and Sampling test in LIMS.

The SC:

G. Reviews any deviation request.

H. Approves or denies deviation from the ESP.

7524.1.2 Exemplar Exhibits

NOTE: 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 randomly select nine units.

B. Analyzes each selected unit per 7525.

C. Evaluates results per 7524.2

D. Forms a composite when directed per 7524.3.

7524.1.3 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 7525.

7524.1.4 Arbitrary Sampling

NOTE: Arbitrary sampling procedures apply whenever sections 7524.1.1 through 7524.1.3 are not followed. When arbitrary sampling is used, no population inference can be made.

The LD:

A. Ensures arbitrary sampling is only used in extenuating circumstances.

The SC:

B. Obtains written concurrence from the customer and the Laboratory Director.

C. Documents communication in LIMS.

D. Documents approval of the deviation from the ESP in the Description of Exhibit and Sampling test in LIMS.

The FC:

E. Ensures supervisory approval is documented in the Description of Exhibit and Sampling test in LIMS.

F. Selects at least one unit and designates it as sub-exhibit X.01.

G. Analyzes the selected unit(s) as directed in 7525.

H. Forms a composite when directed as in 7524.3.

I. Separates remaining units as sub-exhibit (e.g. X.02) for no analysis.

1. If net weight and unit count are obtained, determine both as directed in 7523.

J. Places the following statement in the *Observations, Results, and Conclusions* section of the DEA-113:

"No analysis per [approval source]. Conclusions reported for [List analyzed sub-exhibit(s)] cannot be applied to the unanalyzed units in [List unanalyzed sub-exhibit]."

7524.2 Evaluating the Results

7524.2.1 Controlled Substances or NPS Identified in the Exhibit

The FC:

A. When at least one consistent controlled substance or NPS is identified in each of the individual units selected for testing, reports the following:

1. Controlled substance(s) and/or NPS identified in all units tested.

a. For exhibits with 10 or more units, include statement referencing a 70% population inference at a 95% level of confidence according to the hypergeometric distribution.

2. Any additional controlled substances and/or NPS identified in some, but not all units selected for testing:

a. Report the number of units the additional controlled substances and/or NPS were identified in.

i. A population inference cannot be made for these additional substances.

3. Any non-controlled substances identified in the exhibit.

B. When one or more units tested do not contain at least one consistent controlled substance or NPS (i.e. a negative result), evaluate to determine if exhibit can be separated into populations based on visual differences or packaging and resample per 7524.1.1. Otherwise, proceed as follows:

1. **1 – 9** Total Units:

a. Create sub-exhibits as needed based on the controlled substance(s) or NPS identified.

b. Determine the net weight of each sub-exhibit.

c. Report substances present in each sub-exhibit per 7524.2.1.A.

2. **10 – 30** Total Units:

a. Analyze all remaining units.

i. When analyzing all remaining units in a tablet exhibit, photograph the front and back of an intact unit prior to analysis.

b. Create sub-exhibits as needed based on the controlled substance(s) or NPS identified.

c. Determine the net weight of each sub-exhibit.

d. Report substances present in each sub-exhibit per 7524.2.1.A.

3. **31 or More** Total Units:

a. Randomly sample and analyze 21 additional units, for a total of 30 analyzed units.

b. Do not sub-exhibit

c. Determine the number of units containing each controlled substance(s) and/or NPS identified.

i. For example:

- *Number of analyzed units containing fentanyl:* 2
  Number of analyzed units containing heroin: 28

  OR

- *Number of analyzed units containing heroin and fentanyl:* 14
  Number of analyzed units containing tramadol: 16

  OR

- *Number of analyzed units containing heroin:* 5
  Number of analyzed units containing fentanyl: 5
  Number of analyzed units containing tramadol: 20

d. Report substances present according to 7524.2.1.1.

7524.2.1.1 Making Inferences on a Population when Negative Results are Encountered

The FC:

A. For exhibits where 26-29 of the 30 analyzed units contain a consistent controlled substance or NPS (fewer than five negative results), reports the following:

1. The consistent controlled substance(s) or NPS identified in the 26-29 positive units.

a. Include statement referencing a 70% population inference at a 95% level of confidence according to the hypergeometric distribution.

2. Any additional controlled substances or NPS identified.

a. Report the number of units the additional controlled substances and/or NPS were identified in.

i. A population inference cannot be made for these additional substances.

3. Any non-controlled substances identified in the exhibit.

B. For exhibits where 5-25 of the 30 analyzed units contain a consistent controlled substance or NPS, reports the following:

1. All controlled substance(s) or NPS identified in the exhibit.

a. Report the proportion of the population (in number of units) containing each substance and its associated margin of error for the controlled substance or NPS contained in more than 5 units in the *Observations, Results, and Conclusions* section on the DEA-113 (see Exhibit 6/7530).

i. Use the population proportion blank form posted to the SFDCC and attach in LIMS on the Population Proportion LIMS test.

ii. A population inference cannot be made for controlled substances or NPS contained in fewer than 5 units.

b. Report in the identification statement in the *Exhibit Analysis* section on the DEA-113 the number of units the controlled substances and/or NPS were identified in (see Exhibit 6/7530).

2. Any non-controlled substances identified in the exhibit.

C. For exhibits where a controlled substance or NPS is identified in only 4 or fewer of the 30 analyzed units and the remaining analyzed units contain only non-controlled substances or have no reportable analytes, report results following arbitrary sampling policy per 7524.1.4.

1. Written concurrence from the customer and the Laboratory Director is not required.

7524.2.2 No Controlled Substances or NPS Identified in the Exhibit

The FC:

A. When at least one consistent non-controlled substance is identified in each of the individual units selected for testing, reports the following:

1. Substances identified in all units tested.

a. For exhibits with 10 or more units, include a 70% population inference at a 95% level of confidence according to the hypergeometric distribution.

2. Additional non-controlled substances identified in the exhibit.

B. When no consistent non-controlled substances can be identified, reports the name of each non-controlled substance identified in the exhibit.

C. When no reportable analyte(s) are identified in each of the individual units selected or data is insufficient for identification, reports “No Controlled Substances”.

7524.3 Sampling to Form Composites

The FC:

A. Samples to form a composite according to Exhibit 7/7524 when additional analysis is required to fulfill the following requirements:

1. Identification of substances when all identification requirements per 7525.3.1 are not able to be fulfilled during pre-composite testing

2. Determination of salt form of a target analyte

3. Quantitation of a target analyte(s)

4. Determination of the geometric or optical isomer of a target analyte

5. Determination of density

B. Does not sample to form a composite 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. Exhibits that do not require additional analysis.

C. Analyzes the composite per 7525.

7525 QUALITATIVE ANALYSIS

7525.1 General Requirements

The FC:

A. Uses a Standard Operating Procedure (SOP) for analysis, when available.

B. Selects an analytical scheme that meets the minimum analysis and identification requirements in 7525.2 and 7525.3, is appropriate for the compounds identified, and includes a combination of methods in order to minimize limitations in the reported results.

C. Uses validated or verified qualitative methods during casework analysis.

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.

D. Evaluates the data of the unknown for suitability in meeting acceptance criteria in 7525.4 prior to comparison to the known.

E. Bases all conclusions on reviewable data which support the reported identifications. Examples of reviewable data include spectra, chromatograms, photographs, or detailed annotations for color, immunoassay, precipitate, and microscopic tests.

1. Any data or observation which is inconsistent with the identification must be fully explained, investigated further, or the compound cannot be reported.

F. Compares the sample results to data from positive controls (i.e., DEA laboratory reference materials) analyzed under the following conditions:

NOTE: Any timeframes listed below may be further limited by the method verification.

1. For color, precipitate, and microcrystalline tests: Within three months. (See 7512)
2. For immunoassay tests: Contemporaneously (i.e. within 24 hours). (See 7525.4.2)
3. For thin-layer chromatography (TLC): Concurrently. (See 7525.4.3)
4. For gas chromatography GC, LC, and CE: Within a month using the same method and instrument.
    a. Positive control data must meet the acceptance criteria found in 7503.1.1.2.
5. For MS, IR, and NMR: Using the same method and instrument.
    a. Positive control data must meet the acceptance criteria found in 7503.2.1.2.

b. For methods with validated variable acquisition parameters (e.g. DART vs. ESI, ATR vs. transmission), collect positive control data under the same instrumental conditions as the sample.

NOTE: This does not apply to permitted method modifications in Exhibit 3/7503.

G. Includes the following with positive control instrumental data: instrument identifier, reference material unique identifier, and date and method of analysis.

H. Bases identifications on established acceptance criteria per 7525.4.

I. Uses negative controls (blanks) with instrumental and chemical tests to verify that the solvents, reagents, and instruments are free of contamination.

1. Negative controls (blanks) use the same solvent, reagent, or internal standard solution as the qualitative sample.

J. Analyzes a negative control using the same method as the sample under the following conditions:

1. For instrumental tests (except NMR): Immediately prior to the sample.
2. For TLC, color, precipitate, and microcrystalline tests: Concurrently.
3. For NMR and immunoassay tests: Contemporaneously (i.e. within 24 hours).

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: See 7525.4 for specific negative control requirements for individual techniques.

K. Retains untested material for possible reanalysis.

1. In the absence of remaining untested material, retain the remainder of the prepared samples (either dried or as prepared) and any procedural blanks.

7525.2 Minimum Analysis Requirements

7525.2.1 Single Unit Analysis

The FC:

A. Follows the general requirements in 7525.1.

B. Analyzes at least two samplings from pre-composite or composite as determined in 7524.

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

C. Includes a separation technique as part of the analysis.

1. Unless otherwise directed in a SOP, use a general-purpose method.
2. Use an appropriate concentration to ensure the detection of low-level substances.

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

7525.2.2 Multiple Units: Pre-Composite Analysis

The FC:

A. Follows the general requirements in 7525.1.

B. Analyzes at least two samplings from each selected unit.

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

C. Analyzes each unit using a separation technique.

   1. Unless otherwise directed in a SOP, if no composite is to be formed use a general-purpose method.
   2. Use an appropriate sample concentration to ensure the detection of low-level substances.

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

7525.2.3 Multiple Units: Composite Analysis

The FC:

A. Follows the general requirements in 7525.1.

B. Unless otherwise directed in a SOP, if pre-composite testing was performed using a limited-purpose separatory method, analyzes composite using a general-purpose separatory method.

   1. Use an appropriate sample concentration to ensure the detection of low-level substances.

C. Analyzes the composite to fulfill the following additional analysis requirements, as needed:

   1. Identification of substances when all identification requirements per 7525.3.1 are not able to be fulfilled during pre-composite testing
   2. Determination of salt form of a target analyte
   3. Quantitation of a target analyte(s)
   4. Determination of the geometric or optical isomer of a target analyte

      NOTE: For positional isomers and diastereomers, see 7525.3.1.1 and 7525.3.1.2.

   5. Determination of density

7525.3 Minimum Identification Requirements

7525.3.1 Identifying Controlled Substances or New Psychoactive Substances

The FC:

A. Identifies controlled substances or NPS in each selected unit of the pre-composite or single-unit composite, provided that a sufficient amount of material exists and all requirements have been met.

1. If acceptable data is unable to be obtained during initial pre-composite testing for controlled substances or NPS, identification may be accomplished through composite sampling.
2. Pursuing the identification of controlled substance(s) or NPS when the data does not meet acceptance criteria (neither during pre-composite nor composite testing) depends on the nature of the substance (e.g., carfentanil) and the quality of the data.
    a. If acceptable data was not obtained to identify a controlled substance or NPS, indicate the substance(s) in the constituent table of the applicable test in LIMS, with a remark that the substance was not confirmed.
3. If a controlled substance is a naturally occurring alkaloid, precursor chemical, reaction byproduct, or degradation product, the substance is not identified or reported unless it is the only identifiable controlled substance in the exhibit.
    a. If not identified, indicate precursor chemicals in the constituent table of the applicable test in LIMS.
4. In the absence of a controlled substance or NPS, pursue the identification and reporting of precursor chemicals per 7525.3.1.

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.
2. The results from two orthogonal techniques, 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.

C. Determines salt form whenever statutory considerations, sentencing guidelines, or control status may be affected, unless impractical to do so.
1. Salt form determination may be performed on the composite.
2. All salt forms determined are reported.

D. Reports “Inconclusive” for any controlled substance or NPS where all of the minimum acceptance criteria have been met but at least one of the following apply:
1. Reference material(s) is unavailable for a positive control comparison
2. The intact structure of the substance cannot be determined

7525.3.1.1 Determining Positional Isomers

The FC:

A. Determines the positional isomer on each selected unit in the pre-composite.
1. If sufficient material does not exist in each unit, identifies the substance, including determination of the positional isomer, in the composite.

2. Identification of the substance and determination of the positional isomer must be from either pre-composite or composite testing, not from a combination of both.

7525.3.1.2 Determining Diastereomers

The FC:

A. Determines diastereomers whenever statutory considerations, sentencing guidelines, or control status may be affected, unless impractical to do so, on each selected unit in the pre-composite.
   1. If sufficient material does not exist in each unit, identifies the substance, including determination of the diastereomer, in the composite.
   2. Identification of the substance and determination of the diastereomer must be from either pre-composite or composite testing, not from a combination of both.

B. Documents in the case file when more than one chromatographic peak indicates the presence of diastereomers whose stereochemistry is not being distinguished.

7525.3.1.3 Geometric and Optical Isomers

The FC:

A. Determines geometric or optical isomeric form whenever statutory considerations, sentencing guidelines, or control status may be affected, unless impractical to do so.
   1. Isomer determination may be performed on the composite.
   2. Determines optical isomeric form of methamphetamine hydrochloride with a concentration greater than or equal to 80% only when requested.

B. Documents in the case file when more than one chromatographic peak indicates the presence of geometric isomers whose stereochemistry is not being distinguished.

7525.3.2 Identifying Non-Controlled Substances

NOTE: For diluents, see 7525.3.3.

The FC:

A. Identifies non-controlled substances based on the following minimum criteria:
   1. The result from testing at least one sampling.
   2. The results from two orthogonal techniques, 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.

B. When a controlled substance(s) or NPS is present:
   1. Non-controlled substances may be identified in a single unit or the composite.
   2. Additional testing to pursue the identification of a non-controlled substance is not required (e.g. dipyrone).

a. If acceptable data was not obtained to identify a non-controlled substance, indicate the substance(s) in the constituent table of the applicable test in LIMS, with a remark that the substance was not confirmed.

7525.3.3 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.

7525.3.4 Analyzing Residue Exhibits

The FC:

A. Follows the general requirements in 7525.1.

B. Analyzes the exhibit as directed in 7525.2, 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. The results from two orthogonal techniques, incorporating at least one confirmatory technique.

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

7525.4 Use of Qualitative Tests and Techniques and Acceptance Criteria

The FC:

A. Does not use portable instrumentation for the identification and reporting of substances in casework.

1. Portable instrumentation is intended for informational purposes and field use only.

B. Uses the qualitative tests and techniques described in this section to identify controlled and non-controlled substances.

C. Bases identifications on the general acceptance criteria for each test.

1. If multiple substances present in one or more units produce the same positive test result, the results cannot be used in the identification of each substance.
   a. This does not apply to naturally occurring alkaloids, incomplete reactions or sample breakdown, unless they are the predominant substance in the exhibit.
2. If multiple substances present produce different test results and all results are observed, the results can be used in the identification of each substance.

D. 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.

7525.4.1 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.

7525.4.2 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 positive and negative control contemporaneously (i.e. within 24 hours) using the same lot number of immunoassay test as the sample(s).

C. Accepts a result when the result observed for the sample is consistent with the positive control.

7525.4.3 Thin Layer Chromatography

The FC:

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

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.

7525.4.4 Gas Chromatography

The FC:

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

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.
    a. 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.
2. Co-analysis shall not be used to differentiate between compounds that do not have discernible apexes.

C. Accepts a result when a single peak with a clear, non-splitting apex is observed.
    1. For thermally labile compounds, the data generated may still be accepted. An alternative technique or sample preparation (e.g. derivatization) must be used to confirm the intact molecule.

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.

7525.4.5 Liquid Chromatography

The FC:

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

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.
        a. 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.
    2. Co-analysis shall not be used to differentiate between compounds that do not have discernible apexes.

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.

7525.4.6 Capillary Electrophoresis

The FC:

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

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.

   a. 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.

2. Co-analysis shall not be used to differentiate between compounds that do not have discernible apexes.

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 $S/N_{pk\text{-}pk}$ 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.

7525.4.7 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 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 7525.1.

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.

7525.4.9 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.

7525.4.9.1 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 7503.2.1.2.1 (e.g., due to spectral tilting, MS/MS 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.

7525.4.9.2 MS as Separatory

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.

7525.4.10 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 (13C-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.

7526 QUANTITATIVE ANALYSIS

7526.1 General Requirements

The FC:

A. Applies balance minimum net weight requirements (7523.1) to the preparation of all quantitation solutions including the preparation of qNMR internal standard solutions (ISS).

B. Uses only calibrated equipment for volume measurements associated with calibrant solution preparations, including the preparation of qNMR ISS.

C. Uses only Class-A volumetric glassware or calibrated automatic pipettes for volume measurements associated with QC and sample solution preparations.

D. Performs quantitative analyses using validated or verified quantitative methods. (See Exhibit 3/7503 for acceptable modifications).

1. A standardized method must be used, if available.
2. When a standardized method is not available, use a laboratory-validated method.

E. 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, investigated further, or the quantitative value cannot be reported.

F. Equilibrates solutions maintained under refrigeration to room temperature prior to use.

G. Uses the same sample preparation (e.g. base extraction) for all quantitation solutions (samples, QC solutions and calibrant).

H. Uses a negative control to ensure that the instrument and solvent are free from potential carry-over or contamination.

1. Negative controls (blanks) use the same solvent or internal standard solution as the quantitative sample.

7526.2 Determining When Quantitation is Required

The FC:

A. Quantitates the following:
    1. Substances that require the result for statutory considerations or that are proficiency testing samples:
        a. Methamphetamine
        b. Amphetamine
        c. Oxycodone
        d. PCP
        e. Hydrocodone
    2. Substances that are purchased exhibits, free-sample exhibits, or proficiency testing samples:
        a. Cocaine
        b. Heroin
        c. Fentanyl
    3. Exhibits when directed by the Office of Forensic Sciences.
    4. Additional substances when requested require LD approval and concurrence with SFQ and SFL1.
        a. Contact SFQ for method availability.
        b. Contact SFL1 for certified reference material availability.

    EXCEPTION: REDACTED

B. Does not routinely quantitate samples when the primary controlled substance is estimated to be present below the 1% level.
    1. Purity values below the 1% level can be reported when requirements in 7526 are met.
C. Performs quantitative analysis of secondary controlled substance(s) that meet the criteria in 7526 and are estimated to be present at a level of 5% or greater, provided that a sufficient amount of sample is available.
D. Quantitates exhibits over the following thresholds provided sufficient sample remains after testing:

Gross Form	Threshold for Quantitation
Powders and crystalline material	Total Net Weight ≥ 100 mg
Tablets	Total Net Tablet Count ≥ 12 Tablets
Liquid	Total Net Volume ≥ 5 mL
Gummy	≥ 5.0 g per Unit

1. For exhibits below these thresholds or other gross forms, purity testing is not normally performed unless requested by the customer.

7526.3 Preparation of Solutions

7526.3.1 Sample Preparation

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 powders, crystalline materials, and tablets, weighs, at a minimum, the sample amount specified in the table below from the composite.

Purity (%):	Minimum Quantitation Sample 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	

Purity (%):	Minimum Quantitation Sample Amounts (mg)[a] for composites ground, mixed, and sieved to:		
	850 µm (20-mesh):	425 µm (40-mesh):	250 µm (60-mesh):
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		
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.

C. For powders, crystalline materials, and tablets, in the event a sample amount less than specified in 7526.3.1.B is used, reports the purity as non-representative of the exhibit.

D. For other scenarios, weighs the sample amount specified below:

Sample Type	Sample Location	Sample Amount	Representative of Exhibit?
Composites amenable to grinding, but not sieving	Composite	≥ 100 mg	Yes
Liquids and Solutions	Composite	≥ 1 mL	Yes
Gummy	Portions of various selected units	≥ 0.5 g	No

7526.3.2 Calibrant Solutions

7526.3.2.1 Separation and UV/Vis Methods

The FC:

A. Prepares a calibrant solution(s) using a CRM.
   1. For separation methods, prepare at least one calibrant solution.
   2. For UV/Vis methods, prepare at least three calibrant solutions.

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 (i.e., LC and UV/Vis) 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.

7526.3.2.2 qNMR Methods

The FC:

A. Uses an internal standard (IS) that is a CRM.

B. Directly weighs the IS CRM to the sample.
   1. Factor the documented purity of the IS into the final amount of the IS.

   OR

C. Prepares an ISS.
   1. Factor the documented purity of the IS into the final concentration of the ISS.

D. 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.

7526.4 Quality Control Samples

The FC:

A. Uses a QC sample from SFL1, when available, to prepare QC solutions for use as positive controls during quantitative analysis.

   1. If a QC sample from SFL1 is not available for a target analyte, prepare QC samples from the laboratory's reference materials collection.

      a. The target analyte must be a CRM.

   2. Prepare QC samples that meet the following criteria:

      a. Include the target analyte

      b. Include at least one other substance

      c. The composition and purity mimic commonly encountered exhibits

   3. A substitute analyte may be used for the NMR QC sample when limited quantities of reference material are available.

B. Prepares two solutions at target analyte concentrations that are within and 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.

   2. For NMR quantitation, prepares one solution within the working range of the method.

C. Uses QC solutions until they are depleted or the results fall outside of the acceptance criteria. (See 7526.6)

D. Documents QC solution preparation procedures in the Quantitation test or attachments.

7526.5 Quantitative Analysis

7526.5.1 Performing a Quantitative Analysis Using a Separation Method

The FC:

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

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

B. 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. 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.

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

2. The QC solutions must bracket the unknown sample in concentration (high and low) and in time (before and after).

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

7526.5.2 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.
   2. The NMR blank and sample must be prepared from the same deuterated solvent source to ensure the solvent is free from potential contamination.

7526.5.3 Performing a Quantitative Analysis Using UV/Vis

The FC:

A. Establishes a multi-point calibration curve by analyzing each calibrant solution five times (i.e. five readings), representing the original validated/verified linear range.
   1. The multi-point calibration curve can be preserved and does not need to be reestablished during the analysis of subsequent samples.
B. Quantitates an unknown sample using a new or pre-established multi-point calibration curve, a negative control, and a set of bracketing high and low concentration QC solutions.
   1. A negative control sample is analyzed immediately prior to the analysis of each exhibit to ensure that the solvent is free from potential contamination.
   2. Each solution is analyzed five times.
   3. The QC solutions must bracket the unknown sample in concentration (high and low) and in analysis time (before and after).

7526.6 Acceptance Criteria

The FC ensures:

A. Blanks are free of contamination and/or carry-over.
B. QC solutions are within ± 5% relative to the known prepared purity of the QC sample.
C. There are no known selectivity interferences.
D. 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%.
E. For UV/Vis, the RSD of the response (Abs) for the five replicate readings of the unknown sample is less than 2%.
F. For qNMR results, the RSD of normalized response for multiple integrals of the unknown sample is less than 2%.

7526.7 Uncertainty of Measurement Estimates Determination for Quantitative Values

The FC:

A. Calculates the uncertainty of measurement estimate (UME) associated with quantitative values per Exhibit 8/7526.

7526.8 Reporting Quantitation Results and Uncertainty Estimates

The FC:

A. Reports the final purity value as the average of the following provided that the RSD criteria are fulfilled:
    1. For separatory techniques, the injections analyzed
    2. For UV/Vis, the five replicate analyses
    3. For qNMR, the multiple integrals used.
B. If two or more sample preparations or instrumental techniques are used, obtains a final purity value as follows:
    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. The final reported purity result is the mean of all accepted averages.
C. 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).
D. Calculates the UME using the final averaged percent purity value (prior to truncation).
    1. Includes a copy of the completed Uncertainty Calculator in LIMS (SFL1 only).
E. Rounds the final expanded UME to one significant figure using ISO/NIST rounding rules (Exhibit 4/7523).
F. Reports the final purity result in percentage truncated to match the significance of the final reported uncertainty.
G. Reports purity results greater than 100% (e.g., 101%) as 100%.
H. Includes a statement in the *Observations, Results, and Conclusions* section of the DEA-113 when purity results are reported

7526.9 Revising UMEs

SFQ:

A. Reviews and updates the uncertainty estimates for purity determinations every accreditation cycle using cumulative system-wide PTP results and other collaborative data.

7527 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 LOM 73.

7527.1 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 absence of remaining untested material, retain the remainder of the prepared samples (either dried or as prepared) and any procedural blanks.

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. Seals the open end of the evidence envelope.

F. Inspects the integrity of the 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.

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

7527.2 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.

7527.3 Resealing Bulk Exhibits

7527.3.1 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. The amount over the threshold that can be retained is dependent on the exhibit type (e.g. kilobricks, tablets) and at the discretion of laboratory management.

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 7527.1 or 7527.2, 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."
   1. If, due to the amount of material in the exhibit, the exhibit is not separated into threshold and bulk portions, the container code remains "EVD".

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 *Exhibit 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."

7527.3.2 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 threshold portion is not to be separated from the bulk material).

B. Reseals the exterior evidence container(s) as in 7527.1 or 7527.2, 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).

7527.4 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.

7528 REANALYZING EVIDENCE

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

7528.1 Reanalyzing DEA and non-DEA Exhibits Originally Analyzed in LIMS

The SC:

A. Reopens the exhibit in LIMS.

   NOTE: Return exhibit from storage or permanent transfer, if applicable.

B. Reroutes the exhibit to Chemistry.

C. Sends the exhibit for analysis.

D. Assigns the exhibit to the FC.

7528.1.1 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 the Exhibit Description 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 as applicable to the request to the newly created sub-exhibit.

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 7527.1 or 7527.2.

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).

7528.1.2 Reporting

The FC:

A. Creates a supplemental report reflecting the reanalysis information. (See 7530.8)

B. Includes the following statement in the *Observations, Results, and Conclusions* section of the DEA-113:

   "Supplemental report to reflect reanalysis. Refer to original laboratory report dated mm/dd/yyyy."

7528.2 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.

7528.2.1 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 7527.1 or 7527.2.

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).

7528.2.2 Reporting

The FC:

A. Creates a supplemental report reflecting the reanalysis information. (See 7530.8)

B. Includes the following statement in the *Observations, Results, and Conclusions* section of the DEA-113:

   "Supplemental report to reflect reanalysis. Refer to original laboratory report dated mm/dd/yyyy."

7529 REMOVING SAMPLE FOR DEFENSE ANALYSIS OR SPECIAL ANALYSIS REQUESTS, 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 or special analysis requests as described below.

NOTE: Special analysis requests may be made by the customer (e.g. THC quantitation) or SFL1 (e.g. reference material).

C. Reweighs evidence as described below.

D. Assesses evidence returned from court as described below.

7529.1 Removing Samples(s) for Defense Analysis or Special Analysis Requests 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.

7529.1.1 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 the sample(s) for 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.
2. For special analysis request samples, label each unit with a unique unit identifier corresponding to the unit sampled.

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 new 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 or Special Analysis Request} 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" (DFA) or "Special Analysis (to SFL1)" (SPA) 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 7527.1 or 7527.2.

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 to vault.

7529.1.2 Reporting

The FC:

A. Creates a supplemental report reflecting the removal of the sample. (See 7530.8)

B. Ensures the supplemental report reflects the new reserve weight.

C. Annotates the Remarks of the *Exhibit Details* section of the DEA-113:

"_____ grams removed for defense analysis." or "___ grams removed for special analysis request."

D. Includes the following statement in the *Observations, Results, and Conclusions* section of the DEA-113:

"Supplemental report to reflect removal of sample for {Defense Analysis or Special Analysis Request} and revised reserve weight. Refer to original laboratory report dated mm/dd/yyyy."

7529.2 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.

7529.2.1 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 7527.1 or 7527.2.

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.

7529.2.2 Reporting

The FC:

A. Creates a supplemental report reflecting the removal of the sample for defense analysis. (See 7530.8)
   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 *Exhibit Details* section of the DEA-113:

   "_____ grams removed for defense analysis."

E. Includes the following statement in 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.

7529.3 Removing Samples(s) for Defense Analysis or Special Analysis Requests from Non-DEA Exhibits

Refer to 7529.1 for removal of sample for defense analysis or special analysis requests for exhibits that have not yet been returned to the submitting agency.

The SC:

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

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

C. Assigns the exhibit to the FC.

7529.3.1 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 the sample(s) for 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 new unit in LIMS.

1. Use the new exhibit number (e.g., 1-D, 1.01-D, 1-S, 1.01-S, etc.) in the Lab Exhibit field.
2. Annotate the Description as "{Defense Analysis or Special Analysis Request} of Exhibit X", where X is the original exhibit number (e.g., 1, 1.01).
3. Select Place in New Container.
4. Select the Container Code of "Defense Analysis" (DFA) or "Special Analysis (to SFL1)" (SPA) 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 7527.1 or 7527.2.

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 to vault.

7529.3.2 Reporting

The FC:

A. Creates a supplemental report reflecting the removal of the sample for analysis. (See 7530.8)

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 *Exhibit Details* section of the DEA-113: "_____ grams removed for defense analysis." or "___ grams removed for special analysis request."

E. Includes the following statement in the *Observations, Results, and Conclusions* section of the DEA-113:

"Supplemental report to reflect removal of sample for {defense analysis or special analysis request} 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 or special analysis request} 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.

7529.4 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, if used, 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 7527.1 or 7527.2.

7529.5 Assessing Evidence Returned from Court

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

7529.5.1 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 7527.1 or 7527.2.
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.
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 7528.1.1.

7530 THE ANALYTICAL RECORD

7530.1 General Instructions

The FC:

A. Uses the LIMS tests to record all raw data, observations, and calculations at the time they are made.
   1. SFL1 maintains the analytical record as a paper case file (i.e., all raw data, observations, and calculations).

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.
   1. Document whether one or both portions of a hyphenated technique are used.

C. Documents the qualitative method used.
   1. Documents all modifications to the method parameters (e.g. split ratio) in the appropriate test or on the attachments

D. Documents the quantitative method used.
   1. Documents all modifications to the method parameters (e.g. split ratio) in the appropriate test or on the attachments

E. Documents the DEA property inventory numbers of all equipment and instruments used in the Equipment tab of the appropriate test.

F. Captures all weighing events using the balance software.
   1. Ensure weighing data are maintained in the case file.
      a. This includes at least one of the following: xml data file, csv data file, or PDF report.

G. Reports all weights, quantitation results, and uncertainties to the appropriate number of significant figures.

H. Attaches photos or digital images to the specific test in LIMS.
   1. 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.

I. Records reference material unique identifier(s) in the appropriate test or on the attachments.

J. Records traceable equipment unique identifier(s) in the appropriate test or on the attachments.

K. 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.

7530.2 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 and Fingerprint units, adds the following LIMS tests:

1. Applicable REDACTED Weight Test
2. Description of Reserve Evidence
3. Gross Weight After Analysis (REDACTED/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 Exhibit 9/7530.

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).

7530.3 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

7530.4 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. Include the following statement in the *Observations, Results, and Conclusions* section regardless of the number of units:

   "Inconclusive result; identification pending further analysis."

2. Other identified substances in the exhibit are reported per Exhibit 6/7530.

D. Ensures that LIMS automatically populates all fields on the DEA-113, except the Remarks fields. (See 7530.5)

E. Reports identified substances in the following order:

1. Controlled substances in order of abundance, if possible,
2. If multiple salt forms of a controlled substance are determined, report all salt forms individually in order of abundance, if possible.
   a. 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.)

3. Non-controlled substances in order of abundance, if possible.

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 ≤ 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 digits (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 ≤ Volume <1000 mL: Truncate and report to whole (e.g., 325 mL).
4. Volume ≥ 1000 mL: Truncate and report to four digits (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. Less than 100 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

L. Submits completed DEA-113 for review.

7530.4.1 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 7530.7.

D. Prepares a DEA-113 to document when there was "No Analysis Performed" on enforcement evidence.

E. Submits completed DEA-113 for review.

7530.5 The Laboratory Report (DEA-113) – Remarks

The FC:

A. Utilizes standardized statements available through the "Insert Phrase" options in Examiner Reports Management (Exhibit 6/7530).

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. All net weight statements.
2. Total unit count and volume, including if total dosage unit count was extrapolated
3. Purity statement.
4. Purity equivalencies (e.g., mg/unit or mg/mL)
5. Remarks for No analysis, Storage only, Supplemental, or Amended reports
6. For inter-laboratory transfers, remark for the location where analysis was conducted.
7. Population proportion statement

D. Enters statements in the *Exhibit Details* section to document the following, as applicable:

1. REDACTED
2. Bulk evidence separation
3. Friction ridge 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:

1. Rule 16(a)(1)(G) Summary of Testimony
    a. For a DEA-113 that is prepared using the blank form instead of LIMS, a separate Rule 16 Summary of Testimony may be used.
    b. For scenarios where the Rule 16 Summary is unable to be used in LIMS, a separate Rule 16 Summary of Testimony may be used.
2. Certificate of compliance statements, as applicable

G. Ensures the Department of Justice (DOJ) Uniform Language for Testimony and Reports (ULTR) reference statement is on the DEA-113.

7530.6 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 for technical and administrative review.

D. Corrects any discrepancies identified by the reviewer.

The Reviewer:

E. Conducts a technical and administrative review of the Case Details Report, the supporting data, and DEA-113 for accuracy, ensuring that:

1. Case, exhibit, and LIMS identifiers are properly documented.
2. Gross weight and evidence descriptions are complete and evidence descriptions are 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.

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.

7530.7 Analyzing and Reporting Foreign Drug Samples (SFL1 only)

SFL1:

A. Analyzes enforcement and REDACTED 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.

7530.8 Revising Laboratory Reports (DEA-113)

The FC:

A. Generates a supplemental DEA-113 when additional results become available (e.g. previous no analysis report) or reanalysis is performed.
    1. Includes the appropriate statement in the *Observations, Results, and Conclusions* section of the DEA-113:

       "Supplemental report to reflect [Insert Reason]. Refer to original laboratory report dated mm/dd/yyyy."
    2. Use the Approved By date from the original report.
B. Generates an amended DEA-113 when errors on the original report are corrected.
    1. Includes the appropriate statement in 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."
    2. Use the Approved By date from the original report.

7541 FIELD ASSISTANCE

7541.1 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 REDACTED.

7542 CLANDESTINE LABORATORIES

The 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.

7542.1 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.

7542.2 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.

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 7522.5)

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

7542.3 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.

7542.3.1 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.

7543 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.

7543.1 Ion Mobility Spectrometer

7543.1.1 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.

7543.1.2 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.

7543.1.3 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

7543.1.4 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.

7543.1.5 Collection Filters

The FC:

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

7543.2 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 (7522.5).
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 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.).

7543.3 Submitting Evidence

The FC:

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

7543.4 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.

7543.5 Reporting Results

The FC:

A. Reports the results on the DEA-113 after laboratory analysis. (See 7525)

NOTE: A separate narrative report is not issued.

7544 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:

7544.1 Plant Material

7544.1.1 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.

7544.1.2 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.

7544.2 Powder Chemicals

A. Submit all material to the laboratory.

7544.3 Retail Packages

7544.3.1 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.

7544.3.2 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.

7544.3.3 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 7544.5 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.

7544.4 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.

7544.5 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

7561 FRICTION RIDGE PROGRAM

7561.1 Separating and Preserving DEA Friction Ridge Print Evidence

The FC:

A. For units opened for analysis, carefully separates all packaging from the drug substance, removing as much drug substance as possible, leaving little or no residue.
    1. If requested by the customer, separate packaging from additional units.

B. While conducting the separation, wears gloves and handles the physical evidence carefully to preserve any friction ridge prints that might be present.

C. Places the separated friction ridge print evidence into an additional container (e.g., plastic bag).
    1. Ensure 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 friction ridge print evidence.

E. Places additional container into an evidence container.

F. Creates a Fingerprint Unit (FIN) in LIMS and adds the LIMS tests listed in 7530.2.
    1. In the Description of Reserve Evidence test, document the number of units for which packaging is submitted for friction ridge print evidence.

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., Friction Ridge Print Examination).

I. Identifies samples containing hazardous substances or objects (e.g., LSD, fentanyl and fentanyl related-substances, drug paraphernalia or biological hazards).
    1. Clearly mark the evidence container as containing a hazardous substance.

2. For evidence sent to another laboratory for friction ridge 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 friction ridge print evidence to the vault.

L. Includes the following statement in the *Exhibit Details* section of the DEA-113:

"Original packaging submitted for friction ridge print examination."

7561.2 Preserving Friction Ridge Evidence Not Separated from Drug Evidence

In some cases, the drug substance present on material to be examined for friction ridge prints cannot be removed without destroying friction ridge 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 friction ridge print processing are needed.

B. Contacts a Friction Ridge Examiner (FRE) to determine the best way to handle and process the evidence if both examinations are needed.

C. Annotates the evidence container according to 7561.1.

7561.3 Separating Friction Ridge Evidence for Other Agencies

The FC:

A. Reviews the submitted paperwork REDACTED for a friction ridge print examination request.

1. If it is impractical to separate other agency friction ridge print/drug evidence, laboratory management will determine if and how to preserve the friction ridge print evidence, or whether the friction ridge print evidence will be examined at the DEA laboratory.

B. Carefully separates packaging from the drug substance as in 7561.1.

1. For packaging being returned to the other agency for friction ridge print examination, separate packaging from all units.

C. Creates additional units in LIMS.

1. Create Fingerprint Units (FIN) for containers being returned to the other agency for friction ridge print examination and route to CT.

2. Follow procedures in 7561.1 for evidence being analyzed by DEA and 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 originating agency for friction ridge print examination."

7561.4 Sampling for Friction Ridge Print Examination of Bulk Drug Evidence Seizures

The Laboratory Director (LD) or designee:

A. Determines friction ridge print examination procedures for bulk seizures (i.e., the number of units to be examined), in consultation with the FC, Friction Ridge Supervisor (FRS), and FRE and an appropriate enforcement official on a case-by-case basis.

7561.5 Drug Evidence Packaging - Bench Transfers

The FC:

A. Follows the bench transfer procedure below when the friction ridge 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 FRE.

   2. Transfer the evidence to the FRE in LIMS.

   3. After friction ridge print examination and reassignment of the exhibit, receive the evidence from the FRE in LIMS.

B. Upon completion of the friction ridge print examination and chemical analyses, reseals both the drug and original friction ridge print evidence.

C. Annotates the Remarks in the *Exhibit Details* section of the DEA-113:

   "Original packaging submitted for friction ridge print examination."

REDACTED

REDACTED

REDACTED

REDACTED

REDACTED

REDACTED

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REDACTED

Exhibit 1/7501 DRUG ANALYSIS DEFINITIONS

Term	Definition
Accuracy **(ASTM E177-20)**	The closeness of agreement between a test result and an accepted reference value.
Adulterant	A non-controlled but pharmacologically active substance that may be added to a controlled substance.
Analogue **(21 U.S.C. § 802)**	Except as provided in subparagraph (B), the term "controlled substance analogue" means a substance: the chemical structure of which is substantially similar to the chemical structure of a controlled substance in schedule I or 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 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. Such term does not include: a controlled substance; any substance for which there is an approved new drug application; 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 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 (SWGDRUG IIIB.1)	An analytical scheme is a combination of selected techniques used to reach a scientifically supported conclusion.
Bias (ASTM E177-20)	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	Seized drug evidence submitted to a DEA laboratory whose net weight exceeds the threshold amount.
Bulk portion	Amount of seized drug evidence in excess of the appropriate threshold amount.
Calibrant	A solution with a traceable purity prepared from a certified reference material (CRM) used to establish a calibration curve.
Calibration Curve	The mathematical relationship that exists between the analyte concentration or sample amount and the signal produced by an analytical technique, over a selected range of concentrations.
Case Details Report (CDR)	The summary of analytical testing and analyst's notes within LIMS.
Certified reference material (CRM) (ISO 17034:2016(E))	Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a reference material certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.
Co-analysis	Simultaneous analysis of an unknown sample and reference material.
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.
Composite	Representative, homogenized material prepared in accordance with the Evidence Sampling Plan and the sampling procedures in Exhibit 7/7524.
Concurrent	At the same time (e.g. in the same sequence)
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, EI-MS, MS/MS, or NMR.

Term	Definition
Confirmed **(see also Identified)**	Used to report the results that fulfill the minimum requirement for an analytical scheme to report the presence of a substance.
Contemporaneous	Within 24 hours
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 k.
Critical resolution pair	For separation analyses, a pair of compounds eluting or migrating with a baseline resolution between 1.5 and 5.0.
Determined	Used to report the results when one test was used to obtain information (i.e., salt form, purity, isomer).
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 (U)	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 also Confirmed)**	Used to report the results that fulfill the minimum requirement for an analytical scheme to report the presence of a substance.
Inconclusive	Results that do not meet criteria for reporting,
Increment	Randomly chosen portion from the exhibit from which the composite is assembled.
Investigating Agency (IA)	The law enforcement agency submitting the physical evidence to the laboratory.
Laboratory exhibit number	A LIMS specific field, which directly relates to the IA exhibit number, used to designate laboratory created sub-exhibits.

Term	Definition
Limit of Detection (LOD)	An estimate of the lowest analyte concentration or sample amount that can be reliably differentiated from the blank matrix and identified by the analytical method.
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.
LIMS case number	A unique identifier which refers to a single IA exhibit.
Linearity **(SWGDRUG)**	The ability of a method to produce test results proportional to the concentration of analyte or sample amount.
Low-response compound	Compound that produces a low-intensity signal under routine experimental conditions.
Measurand	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	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 **(ISO/IEC Guide 99:2007)**	Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand.

Term	Definition
Method	The technique (e.g., gas chromatography, color test, infrared spectroscopy) and associated operating parameters, reagent preparations, sample preparation steps, and data evaluation steps that are 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.
New Psychoactive Substance(s) (NPS) **(Modified UNODC definition; Ther Adv Psychopharmacol 2020, Vol. 10: 1-21)**	Substances of abuse, either in pure form or a preparation, that are not controlled, but which may pose a public health threat. NPS include synthetic stimulants, synthetic cannabinoids, synthetic hallucinogens and synthetic opioids and benzodiazepines.
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.
Performance Characteristic **(Bliesner, David M. Validating Chromatographic Methods, John Wiley & Sons, Inc., 2006.)**	Characteristics of an analytical method that define its performance such as accuracy, precision, selectivity, range, etc.

Term	Definition
Physical evidence	May consist of drugs, chemicals, laboratory equipment, packaging, photographs, documents, friction ridge 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.
Precursor chemical	A raw material for a controlled substance that becomes part of the finished product.
Presumptive test	Analytical test that provides an indication of the sample composition.
Probe technique	A procedure whereby units are pierced or small openings are made and a small amount of material is removed for analysis.
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 (e.g. the solvent for a separation technique,) which has been taken through every step of the analytical protocol using the same glassware, sampling implements, reagents, solvents, and analytical instrument.
Quality control (QC) sample	A material that is well-characterized 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 material (RM) **(ISO 17034:2016(E))**	A material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Term	Definition
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 (Short-Term Precision)	The degree of agreement among individual test results when the procedure is applied multiple times, over a short time interval.
Reproducibility (Long-Term Precision)	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. Separation may be based on time or mass.
Standard uncertainty	Measurement uncertainty expressed as a standard deviation.
Sub-exhibit	The separation of an exhibit into two or more sub-sets of the original exhibit.
Target analyte(s)	Substance(s) to be identified (qualitative analysis) or measured (quantitative analysis).
Technique	Wet chemical or instrumental tests that provide information about the composition of a substance.
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.
(Continued below)	Cocaine base: 10 kg of a mixture or substance containing cocaine base.

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 The Merck Index. 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, 3rd 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	A numeric or alphanumeric string that is associated with a single entity within a given system. [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 linearity, LOQ, repeatability, and accuracy for the validation of a given method.

Exhibit 2/7501 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 friction ridge program can be found in LOM 7700, while abbreviations for the digital evidence program can be found in LOM 7600

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
CAFF	Caffeine
CBC	Cannabichromene
CBD	Cannabidiol
CBG	Cannabigerol

Compounds	
CBN	Cannabinol
COC	Cocaine
DMP	Dimethyl Phthalate
DMSO2	Dimethyl Sulfone
DMTP	Dimethylterephthalate
EPHED	Ephedrine
HER	Heroin
METH	Methamphetamine
MJ	Marijuana
NN-DMA	N,N-Dimethylamphetamine
OXY	Oxycodone
PDMAC	para-Dimethylaminocinnamaldehyde
pFF, oFF, and mFF	para-Fluorofentanyl, ortho-fluorofentanyl, and meta-fluorofentanyl
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/Techniques	
CE	Capillary Electrophoresis
DART	Direct Analysis in Real Time

EI	Electron Ionization
ESI	Electrospray Ionization
GC	Gas Chromatography
HPLC	High Performance Liquid Chromatography
IMS	Ion Mobility Spectrometry
IR	Infrared Spectroscopy
LC	Liquid Chromatography
LFIA	Lateral Flow Immunoassay
LTM	Low Thermal Mass
MS	Mass Spectrometry
NMR	Nuclear Magnetic Resonance Spectroscopy
QQQ	Triple Quadrupole
QTOF	Quadrupole Time of Flight
TLC	Thin Layer Chromatography
UHPLC	Ultra-High Performance Liquid Chromatography
UV-VIS	Ultraviolet-visible Spectroscopy

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

Miscellaneous	
B/C	Because
BE	Base extraction or base extracted
Bioh	Biohazard
Bkgd	Background
Bl	Blank
Clan Lab	Clandestine Laboratory
Comp	Composite
Cntg	Containing
REDACTED	REDACTED
Decon	Decontamination
DNMC	Does Not Meet Criteria
Eff	Effervescence
ESP	Evidence Sampling Plan
FC	Forensic Chemist
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
Insol	Insoluble
IO	Insolubles Observed
IS	Internal Standard
ISS	Internal Standard Solution
LQC	Low Quality Control Sample
Macro	Macroscopic exam

Miscellaneous	
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
PBl	Procedural Blank
QNS	Quantity Not Sufficient
QS	Quantity Sufficient
Qual	Qualitative
Quant	Quantitative
Repst	Representative
Rgt	Reagent
RM	Reference Material
RMT	Relative Migration Time
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

Miscellaneous	
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
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

Packaging	
ZPB	Ziplock Plastic Bag

Reagents	
4-AP	4-Aminophenol
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-Cl	alpha-Methoxy-Alpha-Trifluoromethylphenylacetyl Chloride
NaNP	Sodium Nitroprusside
pDMAB	para-Dimethylaminobenzaldehyde
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
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

Solvents	
SolvD	0.4 mg/mL C24 in 9:1 Chloroform:Methanol
103-ISS	0.4 mg/mL Dimethyl Phthalate in 4:1 Chloroform:Methanol
108-ISS	0.4 mg/mL C24 in 1:1 Chloroform:Methanol
THC-ISS-A	0.05 mg/mL 4-androsten-3,17-dione in 9:1 Methanol:Chloroform
THC-ISS-T	0.05 mg/mL testosterone in 9:1 Methanol: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

Exhibit 3/7503 QUALITATIVE AND QUANTITATIVE METHOD MODIFICATIONS

All method modifications must be documented in the casefile or method validation/verification report.

Qualitative Methods:

A. Modifications to qualitative methods that enhance the detection capabilities of the method are permitted without further action. Such modifications include:

1. Decreasing the split ratio.
2. Changing the sample solvent (except NMR).
3. Increasing the injection volume.

   NOTE: Be aware of capacity limitations on GC injection port liners. Lowering the split ratio is recommended over an increase in injection volume for GC methods.
4. Shortening the solvent delay.
5. Extending the hold time at the end of a method.
6. Increasing the number of scans (IR, Raman, and NMR only).
7. Changing the threshold or gain to improve sensitivity.

B. Any modifications made to decrease the sensitivity or to limit the detection of certain analytes require supervisory approval (e.g., extending the solvent delay to intentionally not detect a specific analyte such as acetaminophen).

C. Trimming a column requires performance verification procedures to be completed per 7506.

1. Positive controls may need to be reanalyzed during case analysis if retention times are outside the acceptance window.

D. Replacement of the column with another column with the same properties and dimensions requires a check of each validated and verified method.

1. The method check includes:
   a. A single injection of a solution meeting the requirements for repeatability testing during method validation (7503.1.2.1)
2. Evaluates the data for each compound tested using the following criteria:
   a. A single peak with a clear, non-splitting apex is observed.
   b. Peaks have minimal fronting/tailing.
   c. The first eluting compound has an absolute retention time $\geq 2t_o$.
      i. For methods validated prior to March 16, 2020 with an established minimum acceptable retention time $< 2t_o$, ensure the earliest-eluting compound is detected.
   d. A minimum $S/N_{pk\text{-}pk} = 3$ is observed, including the 0.5% low-level marker compound.

3. Accepts the results 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.
4. Documents the check, results, and technical review in the instrument logbook.

Quantitative Methods

E. The following modifications to quantitative methods are permitted without further action provided all solutions (i.e. blank, calibrant, QCs, and samples) are analyzed using the same method parameters:
   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.

F. The following modifications require a check of the quantitative method:
   1. Replacement of a column with another one having the same properties and dimensions.
   2. The method check includes:
      a. Analysis of the closest eluting 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 (7504).
      d. Documentation of the check, results, and technical review in the instrument logbook.

Qualitative and Quantitative Methods

G. The following changes to qualitative and quantitative methods require an assessment of the applicable performance characteristics required to evaluate in order to establish that the modified method is fit-for-purpose. This may involve additional method verification or method validation testing. Consult SFQ for guidance.
   1. Reducing the run time
   2. Change in gradient (i.e., temperature, buffer ratio, voltage, flow).
   3. Change in detector parameters (i.e., scan rate on MS, resolution on IR).
   4. Installation of a column with different properties, dimensions, or technology.
   5. Implementation of a different buffer composition (e.g., phosphate buffer changed to acetate buffer or pH 3 changed to pH 5).
   6. Change in the scan range.
   7. Change in carrier gas for separatory methods.

Exhibit 4/7523 ISO/NIST ROUNDING RULES

A. 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.

B. 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.

C. 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

D. 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).

Exhibit 5/7524 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.).
   a. For multiple containers containing individual units (e.g. 4 baggies containing tablets), the units should remain segregated and the lottery method applies to each unit for sampling.
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.).

Exhibit 6/7530 REPORTING STATEMENTS

Observations, Results, and Conclusions Section

Net Weight	
Scenario	Reporting Statement
All Exhibits	The net weight represents the weight of all material, excluding the packaging.
All Units - 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.
Extrapolation (in Groups)	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.
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.
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.
Sub-Exhibit - Manually Counted Units	The net weight is an extrapolated value based on the average weight per unit obtained by direct weighing of all submitted units. The net weight uncertainty value represents an expanded uncertainty estimate at the 95% level of confidence.
Exemplars Exhibits	The net weight was determined by direct weighing of all unit(s). No net weight uncertainty reported.
Exemplars Exhibits (Quantitated)	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.

Net Weight	
Scenario	Reporting Statement
Exemplar Exhibits - Different A	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.
Exemplar Exhibits – Same Containers	The net weight is an extrapolated value based on the individual weight of 1 unit. No net weight uncertainty reported.

Population Proportion Statement	
Scenario	Reporting Statement
≥ 5 Negatives *(Include statement for each group)*	Based on population proportion calculations, [Substance identified] is contained in X ± X units at a 95% level of confidence.

Purity	
Scenario	Reporting Statement
*A representative mixture **and** minimum sample amounts are used*	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.
*A representative mixture **and** minimum sample amounts NOT used* *OR* *A non-representative mixture*	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.

Volume and Dosage Unit Count	
Scenario	Reporting Statement
Volume	Total volume = X mL (net); X mL (reserve); substance concentration: Y mg/mL.

Dosage Unit Count	Total dosage unit count = X [Units] (net); X [Units] (reserve); substance concentration: Y mg/[unit].
Additional statement for Extrapolated Dosage Unit Counts	Total number of units is an extrapolated value.

Additional Statements	
Scenario	Reporting Statement
Arbitrary Sampling	No analysis per [Enter approval source]. Conclusions reported for [List analyzed sub-exhibits] cannot be applied to the unanalyzed units in [List unanalyzed sub-exhibit].
Supplemental Report	Supplemental report to reflect XXX. Refer to original laboratory report dated mm/dd/yyyy.
Amended Report	Amended report to reflect XXX. Refer to original laboratory report dated mm/dd/yyyy.
No Analysis Report	No Analysis as per [Enter approval source/reason].
Inconclusive Result(s) Reported	Inconclusive result; identification pending further analysis.
Interlaboratory Transfers	All analyses were completed at the [Laboratory Name] Laboratory, [City, State].

Exhibit Details Section

Friction Ridge Print Examination	
Scenario	Reporting Statement
DEA Exhibits	Original packaging submitted for friction ridge print examination.

Non-DEA Exhibits	Original packaging separated and returned to originating agency for friction ridge print examination.

Additional Statements	
Scenario	Reporting Statement
REDACTED	REDACTED.
Bulk – DEA Exhibits	X grams held for destruction pending written notification.
Bulk – Other DOJ Agency Exhibits	X grams separated in excess of the threshold.
Defense Analysis	X grams removed for defense analysis.

Exhibit Analysis Section

Single Unit Composite Identification Statements for Remarks Section of DEA-113 form:
Single unit composites
A composite was formed from 1 unit for testing. [List Substance(s) Identified] identified in the composite. Salt form [and/or] isomer determined from testing the composite. *No reportable analytes identified or data insufficient for identification:* A composite was formed from 1 unit for testing. No substance(s) identified in the composite.

Constructing Identification Statements for Remarks Section of DEA-113 form (Select parts that reflect analysis performed, as needed)	
Part 1: List substance(s) identified in all units tested. A population inference is made when statistical sampling was performed and a substance is identified in each unit selected for testing.	
A. Positive results for all units, when all units are tested **NOTE**: For single unit composites, see above.	[List substances identified] identified in [#] unit(s) tested.

Constructing Identification Statements for Remarks Section of DEA-113 form (Select parts that reflect analysis performed, as needed)	
B. Hypergeometric Distribution 1. Positive results for all units, 10 or more units 2. 1-4 Negative Results	[List substances identified] identified in X unit(s) tested indicating, to at least a 95% level of confidence, that at least 70% of the units in the population contain the substance(s).
C. No reportable analytes identified or data insufficient for identification for all units	No substance(s) identified in [#] unit(s) tested.
D. Data insufficient for identification in the pre-composite, identification in the composite	[#] unit(s) tested.
E. Population Proportion, ≥ 5 Negative Results	[List substances identified] identified in [#] unit(s) tested.
Part 2: List additional controlled substance(s) and NPS identified in some, but not all of the selected units during pre-composite testing. No statistical population inference is made.	
[List controlled substance(s)] also identified in [#] units tested.	
Part 3: List the number of units used to prepare the composite, when applicable.	
A composite was formed from [#] units for further testing.	
Part 4: List any {additional} substances identified in the exhibit.	
[List Substance(s) Identified] {also} identified in the exhibit.	
Part 5: List salt form and optical isomer determination, when applicable.	
Salt form determined from testing {1 unit/the composite}.	
Isomer determined from testing the composite.	

NOTE: [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).

Weight and Volume PTPs	
Scenario	Reporting Statement
Weight PTP	Analyzed for weight measurement only.
Weight and volume PTP	Analyzed for weight and volume measurement only.

Other

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.

Rule 16(a)(1)(G) Summary of Testimony statement
The following summary of testimony is provided as required by Federal Rule of Criminal Procedure 16(a)(1)(G) and is a complete statement of my opinions, which are exclusive to and address only the exhibit(s) identified in this summary. I am employed by the U.S. Department of Justice, Drug Enforcement Administration (DEA) and was so employed when I conducted the examinations and analyses of the above referenced LIMS number. My qualifications to conduct the examinations and analyses, and to express an opinion as to the identity of the material contained in the exhibit(s) described above, are based on my knowledge, skill, experience, training, and education. See my Curriculum Vitae (which will be provided prior to the close of expert discovery) for additional information regarding my qualifications, including previous testimony offered in the last four years and any publications authored in the last ten years. The opinions described are based on the listed chemical, physical, and instrumental analyses, the results generated by those analyses, and my interpretation of those results set forth in the laboratory report and analyst notes. The manner and process by which I performed the analyses were, to the best of my knowledge, in accordance with the publicly available Analysis of Drugs Manual (ADM) and Laboratory Operations Manual (LOM), in effect at the time of analysis. These are generally available at: https://www.dea.gov/resources/documents?f%5B0%5D=publication_type%3A2596, or were otherwise disclosed upon request. I analyzed the material contained in the exhibit(s) which were submitted for analysis in the above referenced LIMS number. Refer to this laboratory report associated with the subject LIMS number. The analytical methods used in these analyses are validated and verified according to our quality assurance policy to ensure the methods are reliable and fit-for-purpose and the techniques utilized are widely accepted and employed in the scientific and forensic community. Summaries of instrumental methods are available at: https://www.dea.gov/resources/documents?f%5B0%5D=publication_type%3A2596. This report, its attachments, and the referenced documents are not an exhaustive or complete recitation of testimony that I may offer. In addition, I may offer opinions in response to questions posed during trial. Pursuant to Fed. R. Crim. P. 16(a)(1)(G)(v), I, the analyst, approve the foregoing disclosure, and reserve the right to amend as necessary to comply with Rule 16's obligations.

Exhibit 7/7524 COMPOSITE FORMATION PROCEDURES

A. When the formation of a composite is required, one of the following options will be chosen:

1. **Option 1**: The FC combines **all** units in the exhibit.
2. **Option 2**: The FC performs incremental sampling to produce a primary sample (composite) that is representative of the entire exhibit (sampling target).
    a. Unbiased incremental sampling must be followed even when negative units are observed during qualitative testing.

Option 1: Combining all units in an exhibit

B. 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.

C. 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.

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

Option 2: Performing incremental sampling

E. 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, even when negative results are obtained
        i. 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, even when negative results are obtained

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, even when negative results are obtained

NOTE: Selected units containing >1 g of material may have to be sampled more than once.

4. Combine all increments to form the composite.

5. 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.

F. 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, even when negative results are obtained

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.

G. 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, even when negative results are obtained

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.

Exhibit 8/7526 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)
- Varying balance operators
- Balance type
- Balance manufacturer
- Balance readability
- Balance calibration procedures and personnel
- Balance accuracy
- Balance location and operating environment
- Balance long-term performance
- Reference weights used

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.

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

The balance calibration component (u_{bal}) is the uncertainty associated with the calibration of the balance and captures differences in balance types and manufacturers, calibration procedures, reference standard weights, and calibration personnel. The weighing process component ($u_{process}$) is the uncertainty associated with the reproducibility of measurements and incorporates variations resulting from balance location (both intra- and inter-laboratory), environment, operator, and performance verification procedures. The balance accuracy component (u_{acc}) is the uncertainty associated with the accuracy of the reference weights used during the performance verification checks and captures the uncertainty associated with the accuracy of the in-house reference weights used throughout the laboratory system. The number of weighing operations and process by which a weight is obtained are both incorporated into the final expanded uncertainty calculations

The table below lists the system-wide mass uncertainty (u_{mass}) values established for each balance readability used for net weight measurements. Values are calculated using the mean u_{mass} value per balance type plus three standard deviations. These values must be used when

calculating the uncertainty associated with net weights obtained either by direct measurement or by extrapolation.

Readability (g):	u_{mass} (g):
0.1	0.21426
0.01	0.041055
0.001	0.0025756
0.0001	0.00060319
0.00001	0.00037791
0.000001	0.000056368

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 applied to the variable being measured (e.g. 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.). 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.

$$Min.\ weight = \frac{k}{u_{rel}}\ (Median\ SD) \qquad \text{Equation 2}$$

Readability (g):	Minimum Weight (g):
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 7506.3.1. The DEA property inventory number of the

balance is documented in LIMS. All net weight, volume, unit count, and uncertainty calculations are determined using the DEA Uncertainty Calculator within the LIMS *Net Weight* test, and a copy of the completed worksheet is included in the case file.

1.1.1 Determination of UME for Direct Weight Cases

When the net weight of an exhibit is obtained by direct measurement(s), the uncertainty associated with the total net weight (U_{NW}) is obtained by multiplying the combined weighing uncertainty (u_w) by a coverage factor (k) corresponding to a 95% level of confidence (Equation 3). As a conservative estimate, all weighing events are assumed and treated as static processes[1] 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).

$$U_{NW} = u_w \times k \qquad \text{Equation 3}$$

where,

$$u_w = u_1 + u_2 + u_3 + \ldots + u_n \qquad \text{Equation 4}$$

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:

Net weight ± U_{NW} = Net weight ± (u_w x 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.

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

$$u_{avg} = \frac{s}{\sqrt{n}}$$ 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:

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

The total extrapolated net weight and uncertainty for the exhibit are:

Net weight ± U_{NW} = (total # units) (Avg. NW ± u_{NW} • $t_{95\%}$)

(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 and other non-enforcement programs performed at SFL1 are exempt from these procedures.

1.2 Purity

The uncertainty associated with purity measurements is affected by factors including, but not limited to, the following:

- Equipment (Instrumentation, balances, volume measuring apparatus)
- Analyst
- Location (environmental factors)
- Analysis method
- Certified reference material purity uncertainty

- Sample preparation procedures
- Target analyte
- Purity level
- Sample composition

The uncertainty associated with purity determinations is assessed by considering three contributing factors: reproducibility, accuracy (bias), and reference materials. Together, these factors take into consideration the most significant components associated with the total estimated uncertainty.

The reproducibility component (u_R) incorporates the variability of purity results obtained across all nine laboratories and captures differences in analysis method, equipment, location, analyst, target analyte, purity level, sample composition, and sample preparation. The accuracy component (u_{Acc}) represents the bias or systematic error associated with the purity measurements. This component is applicable to all analysis methods, equipment, locations, analyst, target analytes, purity levels, sample compositions, and sample preparations. The certified reference material component (u_{RM}) is the uncertainty associated with the purity value of the certified reference materials used as calibrants during purity measurements.

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:

$$U = \%P \cdot u_c \cdot k_{95\%} \qquad \text{Equation 6}$$

where,

$$u_c = \sqrt{u_R^2 + u_{Acc}^2 + u_{RM}^2} = \sqrt{(0.0702 - 0.012\ln(\%P))^2 + 0.02887^2 + 0.005921^2}$$

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} =	relative uncertainty associated with the laboratory system's accuracy (bias)
u_{RM} =	relative uncertainty associated with the purity of the reference materials used in the laboratories for preparation of the calibrant solutions.

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 QC samples per 7526.4. 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 PTP data are used to evaluate the reproducibility of DEA's quantitative processes. Evaluation of over five years (2018 – 2022) of PTP results indicates that the RSD obtained for quantitative analyses can be estimated by a natural log function of concentration (% purity), with higher RSD values observed as the concentration of the analyte decreases. This behavior is similar to that previously characterized and documented by Horwitz and collaborators[2][3] 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 2018 – 2022. 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 (dashed line) describing the dependence of RSD on concentration, mathematically illustrated by Equation 7.

$$RSD = 0.0702 - 0.012\ln(\%P) \qquad \text{Equation 7}$$

[2] 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.
[3] Boyer KW, Horwitz W, Albert R. Interlaboratory variability in trace element analysis. Anal. Chem. 1985; 57:454-459.

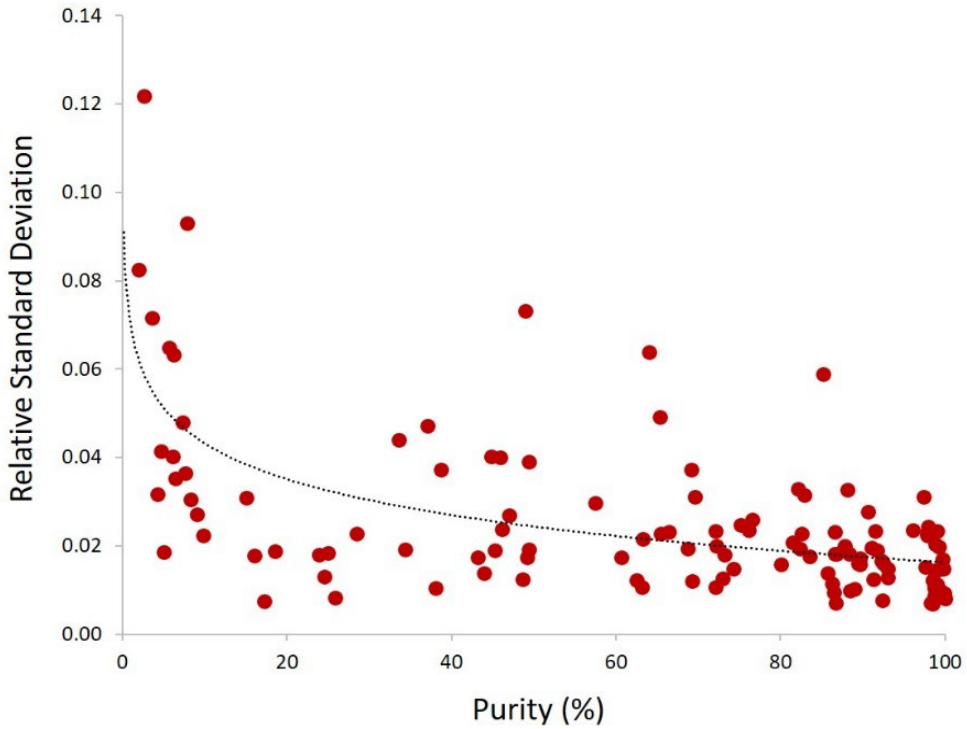

Figure 1: Dependence of PTP RSD values on purity

1.2.2 Accuracy (u_{Acc})

The DEA laboratory system-wide accuracy (bias) uncertainty factor was determined through a Type-B approach and is estimated based on the 5% accuracy acceptance criteria established by DEA policy for method validation by assuming a rectangular (uniform) distribution where the standard deviation is calculated as 0.05 divided by the square-root of 3. This value is applied as a constant relative uncertainty value of 0.02887 across all concentrations.

$$u_{Acc} = \frac{0.05}{\sqrt{3}} = 0.02887 \qquad \text{Equation 8}$$

1.2.3 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). The uncertainty associated with this component was assessed via a type-A (statistically based) evaluation, using the experimentally determined relative uncertainty values associated with recently characterized reference materials and calculating an overall average relative uncertainty. The unexpanded relative uncertainty from 64 quantitative reference materials prepared by the reference materials program at SFL1 were evaluated. The calculated mean of 0.005921 will be used as the u_{RM} component in the purity uncertainty budget as a constant factor across all purities.

$$u_{RM} = 0.005921$$

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:

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

where,

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

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

The final uncertainties associated with purity and amount of pure substance determinations are automatically calculated within LIMS as part of the Summary of Findings test.

Exhibit 9/7530 COMPLETING LIMS TESTS

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. 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 and continued in another space or in an attached document, reference the location of the continued 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 REDACTED 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.

2. For *Gross Form* "Tablet," complete the findings for *Top Logo*, *Bottom Logo*, *Color*, and *Shape*, as follows:

   a. If there are no markings on either side, complete both the *Top Logo* and *Bottom Logo* findings with "*N/A*".

   b. When one side of the tablet has a marking:

      i. Include the marking in the *Top Logo* finding.

      ii. Complete the *Bottom Logo* finding with "*N/A*".

   c. When both sides of the tablet have markings:

      i. Include the alphabetical marking in the *Top Logo* finding (e.g. M).

      NOTE: Include the entire marking if the logo includes both alphabetical and numerical characters.

      ii. Include the numerical marking in the *Bottom Logo* finding (e.g. 30).

   d. When multiple logos are present in an exhibit, complete the *Top Logo* finding with "Multiple logos" and the *Bottom Logo* with "*N/A*".

      i. A description of each logo may be included in the *Remarks* but it is not required.

   e. Do <u>not</u> include:

      i. Quotations,

      ii. Font descriptions (e.g. scripted, italicized, raised, stylized, etc.),

      iii. Scores (i.e. descriptions or simulations of tablet scores using spacing or special characters such as /, \ |, etc.).

   f. Select the predominant color if there are color variations (e.g. speckling).

      i. For a container of mixed tablet colors, select "*Various colors per container*."

   g. For colors or shapes not listed, select "*Other*" and provide a description in the *Remarks* section.

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 to include:

1. The sampling procedure used to select the units for identification, including the random sampling technique (if used),
2. The tests used during pre-composite testing when a composite is formed.

   NOTE: Not applicable for single-unit exhibits that proceed straight to composite.
3. The composite formation (i.e. Option 1 or Option 2) including the random sampling technique (if used)
4. The 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. 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).

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

H. Document the use of the *Legacy Calculator*.

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

C. Document the use of the *Legacy Calculator*.

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

7 Population Proportion Test

A. Add this test when the population proportion blank form is used.

B. Attach the population proportion blank form.

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

9 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.

10 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.

11 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.

12 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.

13 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.

14 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.

15 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.

16 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.

17 Color Tests

A. Uses this procedure for all color tests.

B. Use a set in 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 a set in 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.

18 Precipitate Tests

A. Use this procedure for all precipitate tests.

B. Use a set in 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 a set in 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.

19 Microcrystalline Tests

A. Use this procedure for all microcrystalline tests.

B. Use a set in 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 a set in 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.

20 Immunoassay Tests

A. Use this procedure for all immunoassay tests.

B. Use a set in 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 a set in 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.

21 Thin Layer Chromatography Test

A. Use a 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 a 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.

22 Chromatographic Tests

A. Use this procedure for the following tests: CE Analysis, HPLC Analysis, and GC Analysis.

B. Use a 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 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 $NaOH_{(aq)}$ 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.

23 Hyphenated Tests

A. Use this procedure for the following tests: GC-IRD Analysis, GC-MS Analysis, and LC-MS Analysis.

B. Use a 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 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 $NaOH_{(aq)}$ and extracted into CH_2Cl_2, etc.) in the *Solvent* finding.

E. Record the name(s) of the compound(s) identified and indicated in the *Sample Result* finding.

1. If the retention time is being used towards the identification of a substance, include a remark that the retention time is consistent with that of the positive control.

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

24 DART-MS Analysis Test

A. Use a 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 sets to document individual sample results by setting the *Negative Control Run* finding to "*No*."

1. Enter the name(s) of the compound(s) identified to the *Spectral Result* finding.

C. Document the DART source and LC-MS instrument used in the *Equipment* tab.

25 NMR Test

A. Use a 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 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.

26 Vibrational Spectroscopy Tests

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

B. Use a 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 sets to document individual sample results by setting the *Negative Control Run* finding to "*No*."

1. Enter the sample preparation used (e.g., Direct, $CHCl_3$ Solubles, Acetone Insolubles/$CHCl_3$ 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.

27 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 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.

28 IMS Test

A. Use a 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.

29 Polarimetry Test

A. Use a 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.

30 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. For *Sample* findings, documents the instrumental technique used in the *Instrument Type* finding.

D. For *GC* instrument type, documents the internal standard solution batch number, if applicable.

E. 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.

F. 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%.

G. Enter the appropriate factor in the *Dilution Factor* finding.

H. 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).

I. For *UV-Vis* instrument type, documents the results of the QC High and QC Low solutions.

J. Document the instrument(s) and balance(s) used in the *Equipment* tab.

K. Use the following conversion factors as the multiplier when salt form corrections are required:

Salt Conversion		Factor
Cocaine	HCl → Base	0.8929
	Base → HCl	1.1200
Heroin	HCl → Base	0.8714
	Base → HCl	1.1475
Methamphetamine	HCl → Base	0.8034
	Base → HCl	1.2446
Amphetamine	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
PCP	HCl → Base	0.8696
	Base → HCl	1.1500
BZP	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.

31 Other Notes Test

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

32 REDACTED

A. REDACTED

1. REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

33 Exemplar Weight Removed Test

A. Add this test to the parent exhibit.

B. Enter the total amount of material removed for REDACTED

34 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

35 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

36 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

37 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

38 REDACTED

A. REDACTED

REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

E. REDACTED

39 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.

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

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

42 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.

43 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.

44 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.

a. If the description of the evidence is too long for the provided space and continued in another space or in an attached document, reference the location of the continued description.

3. Record the date the container was sealed in the *Date Sealed* finding.

B. If applicable, document each instance the container(s) was resealed in the *Remarks* finding.

C. If applicable, document threshold or interior packaging weights in *Case Attachments*.

45 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.

46 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.

47 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.

48 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 47-49.

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*".

48.1 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

E. REDACTED

F. REDACTED

G. REDACTED

H. REDACTED

I. REDACTED

J. REDACTED

48.2 REDACTED

A. REDACTED

49 REDACTED

A. REDACTED

B. REDACTED

C. REDACTED

D. REDACTED

E. REDACTED

F. REDACTED

G. REDACTED

H. REDACTED

I. REDACTED

50 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 (APS), 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.
2. *Substance Purity*: Report purity and UME as percent.
3. *APS*: The APS is the product of the reported (truncated) net weight and the reported (truncated) purity.
4. *Reserve Weight*: Report in the same units as the net weight.

REDACTED

REDACTED

END OF DOCUMENT

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