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Abbreviations and Definitions

Version:	Controlled document revision number after SharePoint controlled document migration (October, 2021)
ACS:	American Chemical Society
ACS grade:	A standard of quality for purchased chemicals
Analyte:	The chemical of interest in a chemical analysis
APAP:	Acetaminophen (for *N*-**a**cetyl-**p**ara-**a**mino**p**henol)
ASTM:	Formerly the American Society for Testing and Materials, a standards development organization
ATR:	Attenuated Total Reflectance
BBDCM:	Borate Buffer / Dichloromethane
BSTFA:	N,O-Bis(trimethylsilyl)trifluoroacetamide
CFR:	Code of Federal Regulations
CH2Cl2:	Dichloromethane (aka Methylene Chloride)
Chemical:	A compound or substance purchased for use in chemistry but not meeting the definition of a drug standard
DCM:	Dichloromethane (aka Methylene Chloride)
DIB:	Drug Identification Bible
Expiration Date:	Date after which a chemical or standard should not be used for casework and should be disposed appropriately
FTIR:	Fourier-Transform Infrared Spectroscopy
GBL:	gamma-Butyrolactone
GC/MS:	Gas Chromatography/Mass Spectrometry
GHB:	gamma-Hydroxybutyric acid
HCl:	Hydrochloric acid
ID:	Identification
ILAC:	International Laboratory Accreditation Cooperation
IR:	Infrared, a type of electromagnetic radiation
IS:	Internal standard
Isomers:	Chemicals with the same chemical formula but different arrangements of atoms
ISO/IEC:	International Organization for Standardization/ International Electrotechnical Commission, a standard development organization
JT:	JusticeTrax (the laboratory information system)
LERM:	Laboratory-established reference material
LSD:	Lysergic Acid Diethylamide, a hallucinogen
m/z:	Mass-to-charge ratio, the x-axis in a mass spectrum
Matrix:	The components of a sample other than the analyte
Matrix-matched:	The use of a reference material with a similar chemical composition to the sample to account for the impact of the matrix on the chemical analysis
MeOH:	Methanol
MDA:	Methylenedioxyamphetamine
MDMA:	Methylenedioxymethamphetamine
MMDA:	3-methoxy-4,5-methylenedioxyamphetamine

MSM:	Methylsulfonylmethane (Dimethyl Sulfone)
NA:	Not Analyzed
NaOH:	Sodium Hydroxide
NCSD:	No Controlled Substances Detected per Alaska Statutes
Negative Control:	A quality measure to ensure a positive result is caused by the sample and not the process or equipment involved
NH4OH:	Ammonium Hydroxide
NFPA:	National Fire Protection Association
PM:	Procedure manual
Positive Control:	A quality measure to ensure the process and equipment can detect a substance known to be present
QC:	Quality control
QIFA:	Quantity Insufficient for Analysis
QIFAC:	Quantity insufficient for analysis unless written approval to consume the evidence is provided by the DA's Office
R0 or R1:	Controlled document revision number (e.g., 2020 R0 is the first revision of 2020) prior to SharePoint controlled document migration (October 2021)
Reagent:	A prepared or purchased substance used for chemical analysis
REF:	Refrigerated
Retest Date:	Date after which a chemical or standard is considered expired unless retested by the manufacturer to ensure continued quality
RT:	Room Temperature or GC Retention Time
Sampling Plan:	A statistically-valid testing approach for a subset of items that makes an inference as to the identity of untested items
SD:	Seized Drugs
THC:	Tetrahydrocannabinol
TMCS:	Trimethylchlorosilane
TOC:	Table of Contents
URL:	The address of a webpage
WI:	Working Instructions

Training of Seized Drug Analysts

Initial Training

In order to perform testing in the seized drugs discipline, an analyst must have completed the Seized Drugs Training Manual or demonstrated competency through prior training and experience.

Mentored Casework

After completion of initial training, an analyst will complete a period of mentored casework. This may include continuing to ask questions of more experienced discipline staff, consulting with other staff on the approach for a particular case (e.g., item selection, weight thresholds, extractions, appropriate second tests for identification, etc.), literature review prior to testimony, and observing testimony of other staff, where available.

Methods of performance evaluation that may be used during this period are technical review, proficiency testing, and testimony monitoring. If a performance issue is identified during this time, notify the Chemistry supervisor.

Sample Selection

Item Selection Policy

At a minimum, routine analysis shall include one item of a suspected controlled substance per defendant, per collection date. Items with weighable quantities will be selected before items with residue quantities. Evidence containing syringes will only be tested if results from all other items submitted are negative and only when the syringe contains a visible volume of liquid deemed by the analyst to be of testable quantity. Additional items may be analyzed if:

- No controlled substance is identified in the first item.
- The weight or count (whichever is reached first) is near a legal threshold and there is a potential that testing of additional items will lead to exceeding that threshold.
- The physical characteristics or accompanying documentation of other items lead the analyst to suspect that a controlled substance of a different schedule may be present.

When testing multiple items may lead to exceeding a quantity threshold (see Statutory Weight Considerations), the analyst may communicate with the prosecuting attorney to determine whether they feel the testing is required. This communication will be documented in the case file. If an attorney has not been assigned to the case yet or if an attorney is not giving a timely response to the analyst's inquiry, additional testing does not need to occur before issuing a report. Either of these situations will be documented in the case file.

If during the pretrial process it becomes apparent that items not tested will require testing then, upon re-submission, these items will receive top priority at the laboratory.

Sample Conservation

An unused portion of the original sample will exist to allow for subsequent retesting. In cases where only residue amounts are submitted, a visible residue must be of quantity sufficient for analysis and reanalysis. Testing of invisible residues is up to the analyst's discretion and is considered on a case-by-case basis. While an attempt is made to conserve sample for reanalysis, additional testing performed in the laboratory (such as latent print testing) may destroy any remaining sample.

For cases with pending criminal charges, written approval to consume the evidence must be provided by the District Attorney's Office. Testing of invisible residues requires written approval provided by the District Attorney's Office.

For cases with no pending criminal charges (e.g., death investigation), approval from the submitting agency is sufficient for consuming the remaining evidence.

Approvals to consume evidence will be documented in the LIMS.

Determining Populations

Seized Drug analysts rely on their training and experience, along with information provided by the submitting agency, to determine which items consist of multiple populations and require sub-itemization prior to analysis.

When some items from a population are not analyzed, no assumptions can be made about the identity of those items unless a sampling plan is followed (see Sampling Plan).

- Samples submitted as separate items must never be combined as a single item by the analyst prior to analysis.
- When analyzing items containing multiple bindles or packages that cannot be visualized through the packaging, the appearance of the contents must be verified by the analyst prior to grouping into populations.
- Factory sealed and labeled packages with the same markings can be considered as one population without visualizing the contents.
- Tablets, capsules (licit or illicit), marked blotter paper, or sublingual films with the same logo, color, and shape submitted as one item can be considered as one population.

Quantitative Analysis for THC

THC quantitation must be performed to differentiate controlled marijuana or THC products from exempt products. Quantitative analysis for THC follows qualitative analysis for the identification of *Cannabis* or cannabinoids.

THC quantitation is performed by third party laboratories at this time. Please contact the Alaska State Crime Lab for details regarding this service.

Quantity Determination

Overview of Balances and Reference Masses

Calibrated balances and reference masses are used to accurately determine the quantities of seized drug case samples. The reliability of this determination relies on the use of appropriate equipment, proper handling, regular calibration, and balance performance checks. This section addresses requirements related to these factors.

Selection of Appropriate Equipment Vendors and Types

The type of equipment being used must be fit for purpose. The following is a list of some factors to consider when identifying appropriate equipment makes and models:

- The requirements of the intended application
- Comparison to equipment already in use
- Whether the equipment is supported by current calibration suppliers
- Units of measurement
- Range of measurement (low and high)
- Accuracy/readability (how many decimal places are displayed)
- Precision/repeatability and impact on reported uncertainty
- ASTM Class (for reference masses only)
- Reliability and maintenance
- Ease of use
- Data transfer capability (balances only)
- Brand reputation
- Number of units required (including redundancy)
- Cost and funding source

Refer to the Balance Calibration Technical Requirements and Reference Mass Calibration Technical Requirements sections for technical requirements for each type of equipment.

Suppliers of calibrated equipment used in Seized Drugs must be accredited to ISO/IEC 17025 by an ILAC-recognized accrediting body and their scope of accreditation must cover the calibration type and range performed. If a device is supplied calibrated, a calibration sticker and calibration certificate must be included. Calibration stickers and certificates must meet the requirements outlined in Calibration Sticker Requirements and Calibration Certificate Requirements, respectively.

Communication with Equipment and Calibration Suppliers

Equipment and calibration suppliers must be notified of the laboratory's requirements regarding equipment and calibration specifications, calibration certificates, and calibration stickers to comply with those requirements. It is recommended to develop a notification template for each calibration type to clearly communicate this information to a vendor (example: Appendix 4 - Notification Regarding Calibration of Reference Masses). Equipment and calibration specifications may also be codified into department or statewide contracts.

Refer to the Balance Calibration Technical Requirements and Reference Mass Calibration Technical Requirements sections for technical requirements for each type of equipment. Also refer to Calibration Frequency, Calibration Certificate Requirements, and Calibration Sticker Requirements for the respective information.

Equipment and Calibration Supplier Approval

A Vendor Approval Supply and Services Form must be completed when equipment and/or calibrations are obtained from a new vendor and on an ongoing basis per the Laboratory Operations Manual. Completed vendor approval forms are stored in Chemistry Vendor Approval Forms. Vendor approval represents the ongoing documentation that equipment and calibration suppliers are capable of meeting laboratory requirements.

Calibration Frequency

Reference masses and balances used for casework will be calibrated before first use (for new equipment) and at least annually thereafter by an approved calibration supplier. It is recommended to request calibration suppliers to mark calibrations as valid through the end of the month for ease of scheduling.

Equipment that is permanently out of service need not be calibrated.

Calibration Certificate Requirements

Each supplied calibration must be accompanied by a hard copy or electronic calibration certificate. Balance and reference mass calibration certificates must include the following elements:

- Indication that the calibration was performed to ISO/IEC 17025 requirements
- Calibration supplier accrediting body and certificate number
- Test item description or model number
- Nominal mass or mass range (reference masses only)
- Calibrated device serial number
- Date calibration was performed
- Date next calibration is due (recommended: end of the month)

- Units of grams and kilograms are used
- Condition as of the item as received/assessed
- As Found and As Left results clearly present
- Tolerance/passing limit applied
- Clear indication when As Found testing does not meet specified requirements
- Where adjustment was performed, a clear indication that this was done
- Uncertainty associated with each calibration level/mass
    - Must include coverage probability as a percent and k-value(s)
- Calibration traceability information
- Uncertainty statement including k-value(s)

Calibration Sticker Requirements

Calibration stickers for balances and reference masses must be applied by the calibration supplier. Calibration stickers for references masses are not applied directly to the masses themselves but rather to their containers. Calibration stickers for both these types of equipment must include the following elements:

- Calibration supplier name
- Calibrated equipment serial number
- Date calibration was performed
- Calibration due date

Handling of Calibrated Reference Masses

To protect the integrity of the reference masses, the following safeguards will be followed:

- Masses are always handled using tweezers (preferred) or lint-free gloves.
- When not in use, masses will be stored in their designated container with a calibration sticker applied.
- If damage or out of tolerance is suspected, the mass(es) will be taken out of service and marked as such. Notify the Technical Lead or discipline supervisor. Calibration is required to determine if the reference mass may be returned the device to service.

Balance Calibration Technical Requirements

External balance calibrations for Seized Drugs must incorporate the following elements.

NOTE: The Range of the calibration is the same as the range of weights that may be reported from that model of balance when used in casework.

- Repeatability will be assessed with 5 or more replicates

- Repeatability will be assessed at approximately 5% or less of the maximum load (see below for approximate loads for each balance type)
- Calibration that represents the intended use:
    - Analytical balances (rated ~220 grams)
        - Readability: 0.0001 g [0.1 mg]
        - Range: 0.0100 gram (10.0 mg) to 100.0000 grams
        - Accuracy tolerance: +/- 0.0003 gram or less
        - Approximate load for repeatability: 10.0000 grams
        - Maximum CMC/Expanded Uncertainty from supplier scope at 100 grams: 0.0008 gram (0.8 milligram)
    - Benchtop balances (rated ~2200 grams)
        - Readability: 0.01 g [10 mg]
        - Range: 0.10 gram (100 mg) to at least 1000.00 grams
        - Accuracy tolerance: +/- 0.02 gram or less
        - Approximate load for repeatability: 100.00 grams
        - Maximum CMC/Expanded Uncertainty from supplier scope at 1 kilogram: 0.010 gram (10 milligrams)
    - Large balances (rated ~16100 grams)
        - Readability: 0.1 gram [100 mg]
        - Range: 100.0 grams to at least 8000.0 grams
        - Accuracy tolerance: +/- 0.2 gram or less
        - Approximate load for repeatability: 500.00 grams
        - Maximum CMC/Expanded Uncertainty from supplier scope at 8 kilograms: 0.14 gram (180 milligrams)
- Each balance is also tested for:
    - Eccentricity – tolerance is the manufacturer requirement
        - Analysts are trained to center load balances; therefore, no specific laboratory requirement is established for this test.

Reference Mass Calibration Technical Requirements

External reference mass calibrations for Seized Drugs must incorporate the following elements:

Reference Mass Calibration Technical Requirements

- 2 milligram to 500 gram kit masses
    - Troemner UltraClass tolerances

- 1 kilogram, 2 kilogram, and 5 kilogram individual masses
    - ASTM Class 1 tolerances

After Calibration is Completed

Evaluate Calibration Technical Information

Read each calibration certificate and compare the number of measurements, range, tolerance applied, repeatability load, etc. to the Balance Calibration Technical Requirements or Reference Mass Calibration Technical Requirements, as applicable. Pay special attention to any items that did not meet As Found requirements.

If any required testing results are missing, contact the calibration supplier to resolve the issue. Notify the Technical Lead and/or the Chemistry Supervisor.

If a balance used for casework did NOT meet the As Found requirements for a weight range that is used in casework, follow the procedure for non-conforming work in the Laboratory Operations Manual.

If a balance does not meet the As Left requirements for a weight range that is used in casework (i.e., it was not able to be adjusted to within tolerance), the balance will be taken out of service and marked as such until it can be repaired or replaced. Consult the Technical Lead and/or the Chemistry Supervisor.

If a balance does not meet the As Found or As Left requirements for a weight range that is NOT used in casework, no action is required.

If a reference mass in use for balance performance checks (see Reference Masses Used) did NOT meet the As Found requirements, follow the procedure for non-conforming work in the Laboratory Operations Manual.

If a reference mass used for balance performance checks does not meet the As Left requirements, the reference mass taken out of service and marked as such until it can be repaired or replaced.

If a reference mass NOT used for balance performance checks does not meet the As Found or As Left requirements, no action is required.

Evaluate Calibration Certificates and Stickers

After evaluating the calibration certificates for technical requirements, the calibration certificates and stickers will be reviewed for completeness and other administrative requirements per Calibration Certificate Requirements and Calibration Sticker Requirements.

If a calibration sticker or certificate is found to have errors or missing information, contact the calibration supplier for a corrected version. If this cannot be easily resolved, contact the Technical Lead and/or the Chemistry Supervisor.

Storing Calibration Certificates

Completed balance and reference mass calibration records are stored in the laboratory SharePoint document library:

Seized Drugs Balance Calibration Records

Seized Drugs Mass Calibration Records

Performance Checks

Regular performance checks are conducted on balances in use for casework in between calibrations. Reference masses are not performance checked between calibrations.

All balances used in casework are checked monthly and each week before use utilizing calibrated reference masses. A check standard is also weighed for the ongoing estimate of the measurement uncertainty for each balance type used in casework. The check standard for each balance model is defined in this manual's Check Standards and Equipment appendix.

Any member of the Forensic Chemistry discipline may perform balance performance checks. Balance performance checks are documented on the Balance Performance Check Form which is stored in the Seized Drug laboratory space. Balance performance check results are also entered in the Balance Performance Checks (for current entries) spreadsheet utilized for the ongoing estimate of the measurement uncertainty.

Reference Masses Used

The following masses are used for balance performance checks: 10 mg, 100 mg, 10 grams, 100 grams, 1000 grams, 2000 grams, 5000 grams.

Checks at 8000 grams are performed by combining the 1000 grams, 2000 grams, and 5000 grams masses.

Balance Performance Check Requirements

The laboratory has defined the following acceptable tolerances for balance performance checks performed by the lab:

Mettler Toledo MX2002	+/- 0.02 gram
Mettler Toledo XSR16001L	+/- 0.2 gram
Mettler Toledo XSR204	+/- 0.0005 gram

If a balance does not pass a performance check, an automated internal adjustment may be used to bring the balance back into specification. If this is done, the performance check will be repeated. If the performance check still does not pass, the balance will be taken out of service until repaired and/or calibrated. Notify the Technical Lead and/or the Chemistry Supervisor.

Balance Use in Casework

Identification of the balance used, the balance reading(s), and the weighing technique (e.g., net or gross) are recorded in the case notes. Readings will also be recorded on the report when they are 0.10 gram or greater (when measured on the Mettler Toledo MX2002 model) or 0.0100 gram or greater (when measured on the Mettler Toledo XSR204 model). Readings less than 0.10 gram on the MX2002 balance will be recorded as "weight is below reporting limit" on the report. Items with weights below 0.10 gram may be reweighed on an XSR204 balance (with estimated uncertainty); however, this is not required for routine casework. If re-weighing is performed, both weights must be documented in the notes.

Specific weighing requirements associated with THC quantitation are addressed in the THC and THCA Quantitation Procedure Manual.

A net weight will be determined for all items analyzed, when practical. If the substance is in such a form as to make weight determination impractical, such as a thin film of residue in a pipe, a net weight is not required. If only a portion of sub-items are tested, a gross weight of the unanalyzed sub-items will be recorded in the notes (this is not required for residue-quantity unanalyzed sub-items).

Liquids will be weighed, and the analyst may document an approximate volume in the notes.

When weighing capsules, the content is not to be removed from the capsule for weighing as the capsule is considered part of the sample. The capsule and content together are considered a net weight.

Proper Weighing Techniques

The following weighing techniques may be applied:

- Place material into a tared container and obtain a net weight. This will accommodate most drug samples.
- Weigh material directly (no separate tared container).
- Weigh the original container with its contents, empty the contents, weigh the empty container, and subtract the difference in the two weights (weight by difference). Analyst must show the subtraction in their notes.

- Obtain the net weights of individual items in an item and add the individual weights (weight by summation). Analyst must show the individual weights and summation in their notes. The reported weight is a combined net weight. A combined uncertainty will also be reported (refer to Uncertainty of Measurement).

Statutory Weight Considerations

The following are weights/counts listed in the Alaska Statutes.

Schedule VIA	1 ounce (28.35 grams)

Multiple Units (Tablets, Blotters, Etc.)

Some items consist of multiple units. Examples include individual bags containing substance, tablets, capsules, blotter paper, bindles, and sublingual films. In general, units will be weighed and counted. Where counts are performed, the count will be listed in the item description on the report.

If an analyst has reason to believe an item to contain many units (~500 or more) in a form that makes counting difficult (e.g., small tablets), the analyst has the option to not count the units if a second Chemistry section staff member observes the initial opening and weighing of the item. The name of the Chemistry member witnessing the opening and weighing will be recorded in the case notes. The witness will also add a case activity indicating which item(s) were observed, a description of the item(s), and the weight(s) observed.

If an item that has already been opened is found to contain large numbers of units, a weight and count are still required.

If multiple weights are recorded in a location other than LIMS and are subsequently entered into LIMS, this counts as a data transfer and must be reviewed by the technical reviewer. Refer to Laboratory Operations Manual, Review Components. In this case, it is recommended to simply add a copy of the location onto which the weights were recorded into LIMS as an attachment including the lab case number to assist the technical reviewer. Recording a measurement directly from a balance into LIMS is an observation and not a data transfer.

Extrapolation of Unit Count

For some cases (e.g., cases involving a Hypergeometric Sampling Plan), it may be desirable to estimate the count of many units using the weight. When a unit count has been estimated using weighing of individual units and the whole, the effect of measurement uncertainty will be considered (see References 2.).

Verification of the procedure to estimate unit count from weight has been performed and is available in SharePoint: 2023.10.24 Verification of a procedure to extrapolate unit count by weight.

Four pieces of information are needed to perform extrapolation of unit count from weight: 1. The total weight of all units (as a single weighing), 2. 10 individual unit weights (all recorded on the same calibrated balance), 3. The unexpanded uncertainty of the balance used for the total weight of all units, and 4. The unexpanded uncertainty of the balance used for the 10 individual weights. Weights are also recorded into JusticeTrax.

A locked worksheet is available which will perform all necessary calculations from the manually entered information: Extrapolate Unit Count from Weight Worksheet.xlsx. Once calculations are complete, the worksheet will be printed to a PDF and attached to the case file in JusticeTrax.

A reported extrapolated unit count will list the estimated unit count +/- the expanded uncertainty, the k-value used, and the confidence interval.

Analytical Scheme

The intent of the Seized Drug analysis scheme is to detect and confirm all controlled substances listed in Alaska Statutes Chapter 71 with the level of specificity required by those statutes (excluding when a Sampling Plan is used). When referring to the presence of a controlled substance, "any quantity" as worded in the Alaska Statutes is interpreted by the laboratory as "a quantity that allows for complete testing using the discipline's Analysis Scheme with all positive results meeting their defined acceptability criteria."

Scope of Testing

Presumptive tests include color tests, physical identification of pharmaceuticals, microscopic analysis (plant material only), TruNarc analyzer, and GC retention time comparison.

Confirmatory tests include GC/MS and GC/DiscovIR.

All analyses conducted will include GC/MS.

Cocaine base or hydrochloride may be reported with a corresponding result from the TruNarc analyzer when the other minimum criteria of the Analytical Scheme have been met.

All Alaska-controlled substances not possible from breakdown or decomposition will be identified and reported unless deemed insignificant by the analyst.

Non-controlled substances will also be identified and reported if their presence affects the schedule of a controlled substance identified in the sample. Furthermore, non-controlled substances may be reported when sufficient information for identification is produced during testing.

Justification for not pursuing the identification of a controlled substance must be adequately documented in the case notes.

Samples of suspected marijuana or THC products will have qualitative analysis performed if that sample would have been selected for analysis based on item selection and if there is sufficient material to do so.

When an identified substance is not specifically listed in the relevant Statutes (e.g., a substituted cathinone under Sec. 11.71.150. Schedule IIA. (e) (15)), the analyst will identify the chemical attributes resulting in the control status in their notes.

Minimum Criteria

The minimum criteria necessary to report a controlled or non-controlled substance are two consistent results that include at least one presumptive test or GC/IR AND GC/MS. Each test is conducted from a separate sampling of the item.

The minimum criteria necessary to report "**No controlled substances per Alaska Statutes detected**" are two GC/MS tests from separate samplings of the item. Each sampling will be extracted with a different solvent and/or pH (see Extraction Protocols).

The minimum criteria to identify any cannabinoid is two consistent chemical test results (e.g., Duquenois-Levine and GC/MS).

The minimum criteria necessary to identify Cannabis are 1. microscopic examination documenting cystolithic hairs and clothing hairs on opposite sides of the same leaf and 2. Identification of at least one cannabinoid using two consistent chemical tests.

Cannabis may not be identified in samples that lack sufficient observable macroscopic and microscopic botanical detail (e.g., very fine plant material, liquid extracts, and residue).

Additional Considerations

Similar Compounds and Specificity

For reported substances known to share similar mass spectral or infrared characteristics with other compounds, an additional test capable of further distinguishing the compounds from one another will be used. This will be done regardless of whether the similar compounds are controlled.

Reports shall only be as specific as the techniques used allow. They will not indicate enantiomer (levo/dextro) or diastereomer (pseudo/allo) forms unless the Statute interpretation requires it and the testing used is capable of the differentiation.

Thermal Conversion

Drugs known to undergo conversion when heated to GC inlet temperatures will be differentiated using pharmaceutical markings, TruNarc, or derivatization if required for statute interpretation. Examples include:

Psilocybin → psilocin

GHB →GBL

Clorazepate →nordiazepam

Oxazepam →quinazoline compound

Lorazepam →quinazoline compound

Ketazolam →diazepam

NBOH compounds→2C compounds

Casework Consultations and Documentation

Consultations consist of varying levels of discussion between analysts. Not all discussions rise to the level of consultation that requires documentation. If there is doubt whether interaction has risen to the level of requiring documentation, it will be documented. Analysts may consult at any time, including prior to starting work on a case.

When documenting a consultation, the original analyst will include the following information in their case notes and a case activity (Comm-Other-Chem): the date of the consultation, the name of the analyst consulted, and the outcome. When possible, if the consultation involved determination of identification (i.e., deciding on the final reported result), the consulting analyst will not serve as the technical reviewer for the case.

A consultation will be documented when it involves one or more of the following elements:

- Extraction and detection methods
- Assessment of the suitability of a sample spectrum for comparison
- Significance of spectral features

Technical Deviations

Deviations from technical procedures not specifically permitted by the Seized Drugs Procedure Manual or a related Working Instruction should be pre-approved. Refer to the Laboratory Operations Manual, Deviation Request Form Policy for more information.

Technical deviations that are not pre-approved are considered non-conforming work and are addressed in the Laboratory Operations Manual in the Quality Assurance Review Policy. Refer to the Laboratory Operations Manual for more information.

All technical deviations are reviewed by another competent analyst during case review.

Instrument Maintenance, Quality Assurance, Parameters, and Reference Libraries

Instrument maintenance logs, instrument quality assurance records, and electronic copies of the manufacturers' instrument manuals are stored in the Laboratory's SharePoint document library.

Gas Chromatography (GC, With Mass Spectrometry)

This section applies to the gas chromatography portion of the GC/MS instruments in the Seized Drug discipline.

The specifications for the column used on an instrument will be recorded in the method located in the instrument's maintenance records.

The QC Mixture is purchased from Cayman Chemical (GC-MS Drug Standard Mixture 3, *Item No. 23539*). It is treated as a primary standard. Refer to the Standards, Training Material, and Reference Materials section for more information regarding its handling and control.

GC Maintenance

Assessing the chromatographic performance of the QC Mixture is the primary way to determine if GC maintenance is necessary. However, the following preventative maintenance tasks will be performed at the minimum frequencies outlined below.

Service	Minimum Frequency
Liner Replacement	Every 2 Months
Septum Replacement	Every 2 Months*
Gold Seal Replacement	Every 2 Years
Syringe Replacement	Every 2 Years

**Not applicable if a Merlin microseal septum is installed*

Procedures for performing these services can be found in the instrument manufacturer manuals.

Purge a new column with carrier gas for at least one hour before installing it to the detector or heating it above room temperature.

After trimming or replacing a GC/MS column, the following will occur:

- Column length will be calibrated for all methods used in casework to improve retention time consistency, if possible. This is accomplished by entering the column's measured hold up time (retention time of an un-retained peak) into the Edit Entire Method portion of the instrument software. The software

limits how much a column length may be calibrated. For example, it does not allow adjustments below 27 meters for a 30-meter column.

- Mass spectrometer solvent delay will be reassessed to ensure that the detector turns on immediately after the solvent peak elutes. This can be accomplished by turning off the solvent delay after injecting a solvent blank or running a method with no solvent delay. The updated solvent delay time is entered into each method used in casework.

GC/MS Quality Assurance

On the first working day of the week, the QC Mixture must be analyzed on the GC/MS (using the SCREENGX1.M method) before it may be used for casework. Whenever maintenance has been performed, the QC Mixture must be analyzed to ensure the instrument is working properly. Passing criteria for the QC Mixture are:

- 5 peaks are integrated*
- Largest peak height is 9,000,000 ± 2,000,000 abundance (MSD)
- Alprazolam peak height is greater than 2,000,000 abundance (MSD)
- m/z 334 present in fentanyl spectrum
- Difference in retention time between heroin and fentanyl is 0.200 minutes or greater (calculation is recorded on the chromatogram by the person approving the QC Mixture)

**The QC Mixture manufacturer has indicated that a trace amount of cocaethylene is present in GC-MS Drug Standard Mixture 3, Item No. 23539. If a 6th peak is integrated due to this trace component, the QC Mixture may still be deemed acceptable.*

If the largest peak height is not within 9,000,000 ± 2,000,000, the SCREENGX1 method's gain factor will be adjusted accordingly. The SCREENGHALF method will also be updated with the new gain factor times 0.5 (half that of GX1).

If any other passing criteria are not met, troubleshooting will occur until the problem is resolved. Maintenance will be recorded in the instrument's maintenance log located in SharePoint.

The person running the QC Mixture will ensure that the standard control number of the QC Mixture appears on the approved chromatogram. The person approving the QC Mixture passing criteria will initial and date all pages of the record and place it into the instrument maintenance records in SharePoint (Digitally Archiving Instrument Records).

GC/MS Instrument Parameters

The SCREENGHALF.M and SCREENGX1.M methods are used when analyzing samples for routine casework on the GC/MS. Copies of the method parameters used by each GC/MS are stored in their respective instrument records in SharePoint. Below is a table listing the GC parameters for the SCREENGHALF.M method used in routine GC/MS casework. This method is for a single-column instrument.

GC/MS Injector Parameters	
Solvent A	Methanol
Solvent B	Hexane
Pre-injection Washes	2 Solvent A, 2 Solvent B
Sample Pumps	3
Post-injection Washes	2 Solvent A, 2 Solvent B
Injection Volume (uL)	1
GC/MS Inlet Parameters	
Mode	Split
Split Ratio	100:1
Inlet Temperature I	250
Septum Purge Flow (mL/min)	3
Column Flow (mL/min)	1
GC/MS Oven Parameters	
Initial Oven Temperature I	100
Initial Oven Time (min)	1
Oven Temperature Ramp (C/min)	25
Final Oven Temperature I	325
Final Oven Time (min)	5
Run Time (min)	15

A GC-MS with a backflush module has an in-line pressure control device enabling the function. This may cause some method inlet parameters to vary from those listed. Here is an example SCREENGHALF.M method for a split-column instrument:

GC Backflush Inlet Parameters	
Mode	Split

Split Ratio	**100:1**
Inlet Temperature I	**250**
Septum Purge Flow (mL/min)	**3**
Column #1 Flow (mL/min)	**1**
In	**Front SS Inlet He**
Out	**PSD 2**
Column #2 Flow (mL/min)	**1.2**
In	**PSD 2 He**
Out	**MSD**

The instrument's software is programmed to automatically integrate detected peaks and perform library searches of their mass spectra. The table below lists the integration parameters used by the GC/MS methods.

Integration Parameters	
Integrator	RTE
Parameters File	screen.p
Data Point Sampling	1
Smoothing	Checked
Start Threshold	0.200
Stop Threshold	0.000
Baseline Reset (# points)>	5
If leading or trailing edge <	100.0%, Baseline drop else tangent
Minimum Peak Area	50000
Peak Location	Top
Maximum Number of Peaks	100

Below is a table listing the MS parameters for the SCREENGHALF.M method used in routine casework:

Mass Spectrometer Parameters	
Tune File	stune.u
EMV Mode	Gain (Variable)
Acquisition Mode	Scan
Low Mass	40
High Mass	550
Threshold	750
Sample #	2
Transfer Line Temperature I	270
Source Temperature I	230
Quad Temperature I	150
Standard Tune Parameters	
Tune File	stune.u
Tune Mass 1	69
Tune Mass 2	219
Tune Mass 3	502
Mass 50 Target (0.3 – 5%)	1
Mass 131 Target (20 – 120%)	55
Mass 219 Target (20 – 120%)	45
Mass 414 Target (0.3 – 10%)	3.5
Mass 502 Target (0.3 – 10%)	2.5
69 Abund. Target, counts (1E5 – 2E6)	500000
Peak Width Target, amu (0.4 – 0.8)	0.55
Maximum Repeller (10 – 42.84)	30
Emission Current (10 – 300)	34.6
Maximum Ion Focus (40 – 127.5)	90
Ion for Ion Focus Maximum (1, 2, 3)	3
Ion for Repeller Maximum (1, 2, 3)	2

GC/MS Methods and Method Changes

Temporary changes to split ratios, MS scan ranges, gain factors, and oven temperature programs are allowed, when appropriate. These changes will be documented in the case file(s) for which the temporarily modified method was used.

Permanent changes to GC/MS methods or new, targeted methods must be approved by the Technical Lead or discipline supervisor EXCEPT the following circumstances:

1. Changing solvent delay due to column maintenance
2. Updating the mass spectral libraries
3. Updating the MS gain factor to achieve quality control requirements

When a permanent change is made, a copy of the archived method parameters is marked with an end date and is retained in the instrument records in SharePoint. The following circumstances do not require reprinting of methods; however, it must be noted on the instrument maintenance log in SharePoint:

1. Updating the mass spectral libraries
2. Updating the MS gain factor to achieve quality control requirements

For a targeted method, the changed method parameters will be noted in the case file(s) for which the targeted method was used. Refer to Appendix 2 – Targeted GC/MS Methods for specific method parameters for targeted methods.

Mass Spectrometry (MS)

Gas Chromatography (GC, With Mass Spectrometry) covers the GC component of GC-MS whereas this sub-section specifically covers the MS component.

MS Maintenance

Assessing the results of the standard tune is the primary way to determine if MS maintenance is necessary. However, the following preventative maintenance tasks will be performed at the minimum frequencies outlined below:

Service	Minimum Frequency
Check PFTBA Level	Annually
Ion Source Cleaning	Annually (or As Needed)
Change Rough Pump Oil	Change Every 6 Months[1]
Scroll pump tip seal replacement	Annually[2]

[1]Only applies to systems with oil rough pumps
[2]Only applies to systems with oilless scroll pumps

Detailed procedures may be found in the instrument manufacturer manuals.

MS Quality Assurance

Each week, mass spectrometers must be tuned with the standard tune (stune.u) program before use in casework and after maintenance or cleaning to ensure consistent ion abundance between instruments. Passing criteria for a standard tune are:

- Relative ion abundances of 18 (water), 28 (nitrogen), and 32 (oxygen) to 69 are less than 10%
- The three principal peaks (69, 219, and 502) have acceptable shapes
- The ion abundance of 502 relative to 69 is 2% or greater
- The electron multiplier voltage is less than or equal to 2600 volts

If the passing criteria are not met, troubleshooting will occur until the problem is resolved. Maintenance is recorded in the instrument's maintenance log in SharePoint. The mass spectra of the QC Mixture components will also be reviewed. To be considered acceptable, the mass spectrum of each QC Mixture component must match that of its corresponding reference spectrum. The GC/MS acceptability criteria outlined in the Instrumentation – Data and Analysis section will be used.

Passing tune results will be kept in the corresponding instrument maintenance records with the corresponding weekly QC Mixture results (Digitally Archiving Instrument Records). Automated library search results for the QC Mixture will be included with the chromatogram.

MS Instrument Parameters

MS Reference Libraries

Spectra obtained from analyzing Working Standards may be used when making casework comparisons. When a working standard from a new chemical that has not been previously added to the in-house library is verified, its mass spectrum will be added to the library.

The discipline's in-house library and/or outside libraries from reputable sources may be used during mass spectral library searches. Each library search report will list which libraries were used for each sample analyzed. Libraries will be located on each instrument computer, typically in the following directory: C:\DATABASE\.

Mass spectra obtained from Working Standards will only be added to the discipline's in-house library if they were analyzed using the Standard Tune parameters. Background subtraction will be used before adding a spectrum to the library if column bleed or other interfering ions are present. When entering the compound name into the library entry, the standard control number will be included. New

entries into the in-house library may be consolidated to reduce the frequency of library editing and archiving.

The discipline's in-house library will be named SCDL-YYMMDD where YYMMDD is the date in which that library version was put into use. Whenever a new entry is added, the updated version will be copied to each instrument computer and replace the previous version.

The contents of outside libraries will not be edited; however, if a newer version of an outside library is obtained, it will replace the previous version on every instrument computer. For tracking purposes, the names of outside libraries will include –YYMMDD where YYMMDD is the date in which that library version was put into use.

Backups of current and previous libraries (both outside and in-house) will be placed on the laboratory's SharePoint document library.

Gas Chromatography (GC, With DiscovIR)

This section applies to the gas chromatography portion of the GC/DiscovIR instrument.

The specifications for the column used will be recorded in the method located in the instrument's records.

A liquid nitrogen generator provides the liquid nitrogen for cooling the disk and mercury-cadmium-telluride (MCT) detector.

The QC Mixture is purchased from Cayman Chemical (GC-MS Drug Standard Mixture 3, *Item No. 23539*). It is treated as a primary standard. Refer to the Standards, Training Material, and Reference Materials section for more information regarding its handling and control.

GC Maintenance

Assessing the chromatographic performance of the QC Mixture is the primary way to determine if GC maintenance is necessary. However, the following preventative maintenance tasks will be performed at the minimum frequencies outlined below.

Service	Minimum Frequency
Liner** Replacement	As Needed
Septum Replacement	As Needed
Syringe Replacement	Every 2 Years

** In the manufacturer manual, the liner is also referred to as the "pre-column" in some places.

Detailed maintenance procedures may be found in the GC-IR Training Document stored on the laboratory network drive. Procedures for syringe replacement may be found in the instrument manufacturer manual.

Purge a new GC column with carrier gas for at least one hour before installing it into the detector or heating it above room temperature.

When a new column is installed, the solvent delay will be reassessed to ensure that the detector turns on shortly after the solvent peak elutes. When the solvent front is eluted, there is a delay before the disk position reaches the track position for sample deposition. Take this into account when selecting a solvent delay time to avoid depositing solvent across the disk as it moves to the starting track position. Assessing the solvent delay may be accomplished by injecting a solvent blank with the delay turned off or set to zero. Updates to the solvent delay will be documented in the instrument maintenance log.

GC/DiscovIR Quality Assurance

The QC Mixture will be analyzed on the GC/DiscovIR during any week the instrument is used (using the ScreenS10 method) before it can be used for casework and after performing maintenance. Passing criteria for the QC Mixture are:

- 5 expected components are detected
- Baseline resolution is achieved between all expected components (determined visually)
- Acceptable IR spectra are produced, and library matches for all expected components show clear correlation with the library

If any of the passing criteria are not met, troubleshooting will occur until the problem is resolved. Maintenance will be recorded in the instrument's maintenance log.

The person approving the passing criteria have been met will place their initials and date on all pages of the QC Mixture stored in the instrument records (Digitally Archiving Instrument Records).

GC Instrument Parameters

The ScreenS10 and ScreenS5 methods are used when analyzing samples for routine casework on the GC/DiscovIR. Copies of the method parameters used by the gas chromatograph are stored in the instrument record in SharePoint.

No permanent changes will be made to GC/DiscovIR methods without approval from the Technical Lead or discipline supervisor. When a change is made, a copy of the archived method is marked with an end date and retained in the instrument's record. Temporary changes to split ratios and oven program parameters are allowed, when appropriate. These changes will be documented in the case file(s) for which the temporarily modified method was used.

The GC parameters for the ScreenS10 method are listed below:

Injector Parameters
Solvent A: Methanol
Solvent B: Hexane
Pre-injection Washes: 3 Solvent A
Sample Pumps: 3
Post-injection Washes: 3 Solvent B
Injection Volume (uL): 1
Inlet Parameters
Mode: Split
Split Ratio: 10:1
Inlet Temperature: 250
Septum Purge Flow (mL/min): 5
Column Flow (mL/min): 1
Oven Parameters
Initial Oven Temperature: 100
Initial Oven Time (min): 1
Oven Temperature Ramp (C/min): 25
Final Oven Temperature: 325
Final Oven Time (min): 5
Run Time (min): 15

DiscovIR Infrared Detector

The Gas Chromatography (with DiscovIR) sub-section covers the GC component of the GC/DiscovIR whereas this sub-section specifically covers the DiscovIR detector component.

DiscovIR Maintenance

The Align, Noise, and Polystyrene test are the primary ways in which the Seized Drug discipline determines whether the DiscovIR module is functioning correctly. However, the following preventative maintenance task will be performed at the minimum frequency outlined below:

Service	Minimum Frequency
Rough Pump Oil Change	Every 6 Months

Procedures for performing this service can be found in the DiscovIR Service Manual.

DiscovIR Quality Assurance

Each day before use for casework, the DiscovIR must have the Align, Noise, and Polystyrene tests performed. This is in addition to analysis of the QC Mixture from the GC/DiscovIR Quality Assurance sub-section. Passing criteria for these tests are as follows:

- Align: Detector voltage ≥4 Volts. This value is displayed live on the screen during the Align test.
- Noise: Average value ≤1.5 mAbs (milli Absorbance units). This value is displayed via a notification box after the test is completed.
- Polystyrene: The 1601 cm^{-1} infrared absorbance band has a measured value of 1601 cm^{-1} +/- 2 cm^{-1}. This value is viewed by accessing the Polysytrene.csv (C:\Data\Polystyrene) spreadsheet on the instrument computer. The value is automatically added to this spreadsheet when the Polystyrene test completes.

The test results are documented in the laboratory's SharePoint document library (Digitally Archiving Instrument Records).

If the passing criteria are not met, troubleshooting will occur until the problem is resolved. Any maintenance that occurs will be recorded in the instrument's maintenance log.

DiscovIR Instrument Parameters

The DiscovIR instrument parameters are listed on the chromatogram page (first page of instrument report) produced after each sample analysis and no separate printed method is available. Default settings are as follows:

Default DiscovIR Parameters
Current Solvent Delay/Start Time: See instrument maintenance log
End Time: 15 minutes
Disk Speed: 3 mm/min
Overdeposit: Off

Parameters available for change are: Solvent Delay/Start Time (typically following column maintenance), run End Time (for late-eluting compounds), and the

overdeposit box (causes an injection to deposit on the same track used for the previous injection).

Parameters not available for change are: disk speed.

Important: The End Time in the DiscovIR parameters must match the end time of the oven program from the GC for the data collection time to match the GC run time.

If parameters are temporarily changed for an analysis, the reason for the change will be documented in the case notes.

When the analyst is processing data, the software is programmed to automatically flag peaks above a specific signal-to-noise ratio and produce a sub-file for each including an infrared spectrum and library search. The instrument reports include the sample identifying information, chromatogram, DiscovIR method parameters, IR spectrum for each flagged peak, and one library hit for each IR spectrum.

Spectra obtained from analyzing Working Standards may also be used when making comparisons. When a working standard from a new chemical that has not been previously added to the in-house library is used, its IR spectrum will be added as described in the DiscovIR Infrared Reference Libraries section.

DiscovIR Infrared Reference Libraries

The discipline's in-house library and/or outside libraries from reputable sources may be used during infrared library searches. The library search report will list which library was used for each sample analyzed. Libraries will be located on the instrument computer in the following directory: C:\Data\Libraries of Customer.

Infrared spectra obtained from working standards will only be added to the discipline's in-house library if they have a flat baseline. Baseline correction via Print Workbook may be used to flatten the baseline prior to entry into the library. When entering the compound name into the library, the standard control number will be included.

The discipline's in-house library will be named SCDL-DiscovIR-YYMMDD where YYMMDD is the date in which that library version was put into use. Whenever a new entry is added to the discipline's in-house library, the updated version will replace the previous version.

The contents of outside libraries will not be edited; however, if a newer version of an outside library is obtained, it will replace the previous version. For tracking purposes, the names of updated external libraries will include –YYMMDD where

YYMMDD is the date in which that library version was put into use. The initial external libraries provided with the instrument do not all list dates in the file name. The initial versions of these libraries are archived as described below.

Backups of current and previous libraries (both outside and in-house) will be placed on the laboratory's SharePoint document library.

Carrier Gas Information

All the gas chromatographs use UHP Grade (99.999%) helium as their carrier gas. Compressed gas cylinders are stored on site. Two cylinders are connected to the gas manifold in Gas Closet # 2124 that delivers gas to the instrument room #2117. Extra cylinders are in the Gas Cylinder Room # 1175 on the first floor of the laboratory.

The gas manifold system will alarm when the manifold pressure drops to about 350 psi. Only the in-use cylinder should have the valves open. Close the shutoff valve on cylinders not currently in use, as well as the valve on the unused side of the manifold. Update the magnetic indicator signs on the cylinders as appropriate (in use, empty, full). Ensure a full cylinder is available in the Gas Closet for the next use.

Some GC/MS instruments are equipped with a Helium Conservation Module. The purpose of this component is to conserve helium gas during non-analytical time by switching the gas flow to nitrogen. Nitrogen gas is provided by a nitrogen gas generator. The Helium Conservation Module turns on the flow of nitrogen automatically according to a programmed schedule; however, the schedule will not interrupt a sequence in progress. Nitrogen flow will begin automatically once the sequence has ended. Once the programmed schedule has ended, the gas supply returns to UHP Grade helium.

Digitally Archiving Instrument Records

Many instrument records are generated and stored as electronic documents in SharePoint. Examples of these documents are:

- Current and archived instrument methods
- Current and archived quality control data for:
  - GC/MS
  - GC/DiscovIR
  - FTIR (legacy)
- Instrument and equipment maintenance forms
- Approved instrument methods (e.g., SCREENGX1.M for GC/MS)
- Instrument reference libraries (e.g., in-house MS library)

NOTE: SharePoint requires a valid login. Metadata fields are used to associate instrument records with a specific laboratory discipline and equipment identifier.

Scientific Crime Detection Laboratory Chemistry Records

For instrument records produced or received in hardcopy, copies generated during the previous year will be scanned, checked for legibility and completeness, and placed in SharePoint. Afterward, hardcopies will be destroyed. This will happen no less than once per year. Examples of these types of instrument records include:

- FTIR preventive maintenance records (legacy)
- Reference weight calibration reports
- TruNarc Analyzer weekly quality control check records

Digitally Archiving Instrument Methods

Current and archived instrument methods are saved in SharePoint.

Scientific Crime Detection Laboratory Chemistry Records

Methods saved in SharePoint are identified using the naming convention:

[Method Name] [Method start date] – [Method end date]

Example: SCREENGX1 2021.04.30 – 2021.09.22

For in-use methods, no end date will be present. When a permanent change has been made to a method, a copy of the new, in-use method will be added to SharePoint according to the naming convention but with the method end date blank. The previous version of the method will have the file name edited in SharePoint to add the end date.

Instrumentation – Data and Analysis

Sequence and Data Handling

GC/MS Sequence Table

Acquired data will initially be saved on each instrument.

After the analyst has completed their review of the acquired data, it will be moved to a folder that identifies which year and month in which the data were collected. At the beginning of each month, a new folder will be created for saving that month's data. Instrument data older than three months should be deleted from the instrument computer.

Data file names will be the laboratory case number followed by a differentiating character(s) if multiple samples are from the same case. Negative controls will be written in the same format with the prefix "B" or "b". Drug standard file names will indicate the name of the standard being tested.

The sample field will contain the case number and item number of the sample being tested and "blank" if the sample is a negative control. When drug standards are analyzed, the standard name and control number will appear on the data.

GC/MS Analysis

Each analyst is responsible for ensuring that the instrument has passed all quality assurance requirements before analyzing case samples. Samples will be analyzed using the SCREENGHALF.M or SCREENGX1.M method. A negative control will be analyzed directly prior to each case sample. A negative control is prepared in the same manner (including evaporative concentration, if applicable) as the sample and on the same day. When multiple case samples are prepared in the same manner and analyzed on the same day, the same negative control may be used.

For retention time comparison, the standard must be analyzed using the same oven temperature program, the same column flow parameters, and within 24 hours of the sample.

Reports from each sample injection are electronically printed on the instrument computer. These reports are entered into the appropriate case file in the LIMS.

GC/MS Acceptability Criteria

Ideally, an acceptable blank is one that results in no integrated peaks other than the internal standard. If additional peaks are present, the analyst will either resolve the issue (e.g., reinject the sample and blank) or include justification in the case file as to why the data were still acceptable.

The internal standard should be integrated and of similar abundance to that in the blank. Where this does not occur, the associated data will either be rejected or kept with documented justification (for example: a method from Appendix 2 – Targeted GC/MS Methods was used in relation to a Sampling Plan). When a discrepancy in abundance is observed. The justification will include what likely caused the discrepancy and why the analyst deemed it acceptable. Justification for the use of negative control data is reviewed by another competent analyst.

For retention time comparisons, the integrated retention time of each case sample analyte and the corresponding standard must be within 0.050 minutes for the test to be considered positive.

A mass spectrum obtained from case sample data is deemed suitable for comparison to a known standard (in-house library) or external reputable library when the chromatographic peak is automatically integrated by the instrument software (see GC/MS Instrument Parameters). An analyst may investigate unintegrated peaks to guide testing decisions; however, this comparison may not be used for identification. The significance of m/z values – presence, absence, and abundance – is considered. No prominent ions should be missing from the unknown (evidence) spectrum. For a mass spectral analysis to be considered acceptable, the main ions and the presence or absence of a 'molecular ion' must agree between the unknown and standard spectra. Due caution will be made when a library search results in different compounds with similar mass spectra (see Additional Considerations).

If GC/MS data are rejected, the reason for the rejection will be recorded in the notes. The data will be saved in the case file in addition to the accepted data.

GC/DiscovIR Sequence Table

Refer to the Seized Drug Instrumental Working Instructions for information on location of sequences and data files.

For case samples, the case number and item number of the sample being tested will appear on the data. Negative controls will also indicate "blank". When drug standards are analyzed, the standard name and control number will appear on the data.

GC/DiscovIR Analysis

Each analyst is responsible for ensuring that the instrument has passed all quality assurance requirements before analyzing case samples. Samples will be analyzed using the ScreenS10 or ScreenS5 methods unless otherwise noted in the case.

A negative control will be analyzed directly prior to each case sample. The sample is deposited over the same track as the negative control to demonstrate that the track was clean prior to sample deposition. A negative control is prepared in the same manner (including evaporative concentration, if applicable) as the sample and on the same day. Due to the additive effect of overdepositing and limitations of detector range, use of internal standard in the final extraction solvent is not recommended. When multiple case samples are prepared in the same manner and on the same day, the same negative control may be used.

NOTE: The GC/DiscovIR instrument is not used for retention time comparison.

Reports from each sample acquisition are electronically printed to a folder on the instrument computer. These files are transferred to their appropriate case files in the LIMS.

The case file will include the blank and sample data along with any library or standard spectra used for identification. If GC/DiscovIR sample data are rejected, the reason will be recorded in the notes. The spectra will be saved in the case file in addition to the accepted data.

GC/DiscovIR Acceptability Criteria

Negative Controls

The negative control injected with a GC/DiscovIR sample demonstrates no gross contamination of injection solvent or the disk; however, some small peaks may be present due to the GC septum, GC column, injection solvent, water vapor in the detector, or other factors. The GC/DiscovIR is a confirmatory analysis and not a screening analysis; therefore, non-analyte peaks in the negative control do not impact the infrared spectrum of the analyte and do not require notation.

If the analyst has reason to believe that carryover is occurring, troubleshooting will be performed. Troubleshooting will be documented in the instrument maintenance log in SharePoint.

Use of Internal Standard

When solvent with internal standard is used, an IR spectrum and library match will appear on the report. The internal standard absorbance is expected to be higher in the sample due to overdepositing, although this may not be obvious on the instrument printout due to scaling.

Library Comparisons

The infrared spectrum of each sample is visually compared with that of a known standard or reputable library in addition to the comparison performed in the

software. The significance of absorbance band peaks – presence, absence, and relative intensity – is considered. No prominent absorbance bands should be missing from the evidence spectrum. For an infrared spectral analysis to be considered acceptable, the main absorbance bands and the "fingerprint region" (from about 1500 to 500 cm-1) must agree between the unknown and standard spectra. Due caution will be made when a library search results in different compounds with similar infrared spectra (see Additional Considerations).

In the GRAMS software, library searching produces a mathematical factor representing similarity of the unknown and reference spectra called the "match quality" with a lower decimal value representing more similarity. If the match quality is recorded in another place prior to entry into JusticeTrax, this counts as a data transfer and must be verified by the technical reviewer. Refer to Laboratory Operations Manual, Review Components. In this case, it is recommended to simply add a copy of the location onto which the match quality(ies) was recorded into LIMS as an attachment including the lab case number to assist the technical reviewer. Recording the number directly from the software in JusticeTrax is an observation and not a data transfer.

Reagents and Chemicals

Definition and Quality Requirements

Purchased chemicals are used for reagent and working standard preparation, sample extraction, and cleaning. Examples of purchased chemicals are solvents, acids and bases, and dry/solid chemicals. Purchased chemicals used for casework (including reagent preparation) will be ACS-grade or better. Refer to Standards, Training Material, and Reference Materials for additional requirements for the quality of drug standards.

Storage Conditions and Expiration Dates

Chemical manufacturers specify the storage conditions of chemicals. Chemicals should be stored according to the manufacturer storage conditions, including consideration of chemical hazards (e.g., no storage of oxidizers with flammables, organics, etc.). Refer to the Health and Safety Manual for more information.

NOTE: Common oxidizers include Nitrites (e.g., Nitric acid), Peroxides (e.g., Hydrogen peroxide), Perchlorates (e.g., Perchloric acid), Permanganates (e.g., Potassium permanganate)

Chemical manufacturers may print expiration or retest dates (see Abbreviations and Definitions) on chemical containers. A chemical should not be used for casework beyond the manufacturer expiration date. Additionally, reagents may not have an expiration date later than that of the earliest component expiration date.

Example: A reagent is prepared that typically has a 1-year expiration. The solvent used expires in 8 months. The prepared reagent must also expire in 8 months.

When a chemical expires or reaches the retest date, the chemical manufacturer may be contacted to determine if the expiration date has been extended. If this is the case, the updated expiration date will be written or printed on the chemical container(s) along with the analyst initials and date.

If a chemical manufacturer has not provided a retest or expiration date on the chemical container or documentation, an expiration date will be assigned by the laboratory according to the nature of the material. This expiration date will be written or printed onto the chemical container(s) along with the analyst initials and date. General guidelines are:

Liquid acids/bases: 4 years from date received

Organic solvents: 4 years from date received

Solid acids/bases: 5 years from date received

Other solid chemicals: 10 years from date received

Other liquid chemicals: 5 years from date received

Labeling Requirements

Purchased chemicals will be labeled when received with date received, initials of receiver, and expiration date.

The receiver will ensure that the manufacturer's expiration date, if provided, appears on each container of the chemical. If no manufacturer expiration date is available, the laboratory will assign one according to Storage Conditions and Expiration Dates and apply it to each container of the chemical. When a chemical container is opened, it will be labeled with the date opened and initials of opener.

At a minimum, containers of prepared reagents will be labeled with the reagent's identity, assigned lot number, hazard labels, and the expiration date.

Prepared Reagent Documentation

Most prepared reagent are documented on the Reagent Preparation Form at the time of preparation (excluding the Weber reagent). Reagents documented on the Reagent Preparation Form are assigned a unique lot number for tracking purposes. The lot number format is MMDDYY (the date of preparation) followed by the preparer's initials. Reagents prepared on the same day will have one or more differentiating characters in their assigned lot numbers in addition to the required format. Reagents that expire on the same day they are prepared do not require a Reagent Preparation Form. The preparation of these reagents will be documented in the notes for the case(s) in which the reagent was used.

The Reagent Preparation Form will include:

- Reagent name
- Storage conditions
- Filled out NFPA hazards label
- Recipe used
- Date reagent was prepared
- Assigned reagent lot number and expiration date (refer to Reagent Recipes and Verification Procedures)
- Initials of preparer
- Chemical ingredient lot numbers and expiration dates
- Volume of reagent made
- Positive control name, control number, and expiration date

- Initials of person verifying the reagent (may be the same as preparer or another Seized Drug analyst)
- Positive control result
- Negative control result
- Date the reagent was first available for use in casework

For reagents not requiring a Reagent Preparation Form (e.g., Weber), the following information will be recorded in the case notes:

- Recipe used
- Chemical ingredient lot numbers and expiration dates
- Positive control name, control number, and expiration date
- Positive control result
- Negative control result

Instrumental data used to verify a prepared reagent will be stored with the Reagent Preparation Form. The form will be stored in the Reagent Log Book until digitally archived.

Re-Verifying Prepared Reagents

All the following reagents except for the Marquis reagent, Weber reagent, Borate Buffer, and Internal Standard Injection Solvents will be re-verified each month using the Reagent Check Form. The forms will be stored in the Reagent Log Book until digitally archived.

The Reagent Check Form will include

- Reagent name
- Verification date
- Lot number of reagent and assigned expiration date
- Positive control name, control number, and expiration date
- Positive control result
- Negative control result
- Initials of person verifying the reagent

The expiration dates for reagents with expiration dates >1 month will be tracked in the Reagent Log Book. During monthly verifications, analysts will re-prepare any reagent with an expiration date falling before the next monthly verification.

Documentation of Removal

The date on which a prepared reagent was removed from use will be documented on its Reagent Preparation Form.

Digitally Archiving Reagent Records

On an annual basis, hardcopies of Reagent Preparation Forms, Reagent Check Forms, and any associated instrumental data that was generated during the previous year will be scanned and placed in SharePoint. After ensuring that these scanned records are legible and complete, the hardcopies will be destroyed.

Reagent Recipes and Verification Procedures

The following are commonly used reagents; however, other reagents may be used if they have been shown to be acceptable by the forensic scientific community (e.g., *Clarke's Analysis of Drugs and Poisons*) and perform as expected with documented positive and negative controls. Recipes may be scaled as needed.

Internal Standard Injection Solvent

Ingredients:	0.5 mg/mL tetradecane in GC injection solvent
Verification:	GC/MS has one integrated peak with the mass spectrum of tetradecane.
Storage Conditions:	Room Temperature
Expiration:	2 years

Borate Buffer

Ingredients:	5.4 g NaOH and 20 g boric acid in 500 mL deionized water.
Verification:	pH checked with pH paper (between 9 and 10)
Storage Conditions:	Room Temperature
Expiration:	1 year

Marquis Reagent

Ingredients:	20 mL concentrated sulfuric acid, 20 drops formaldehyde (37%)
Verification:	Positive (guaifenesin – purple OR methamphetamine – orange), negative (blank spot plate)
Storage Conditions:	Room Temperature
Expiration:	1 month

Weber Reagent

Ingredients:	Solution A: Add ~10 mg of Fast Blue B Salt [o-Dianisidine bis(diazotized) zinc double salt] to approx. 2 mL deionized water. The solution will have a faint straw color. Solution B: Concentrated HCl
Documentation:	Record the Fast Blue B Salt lot number and expiration date in the notes for each case in which it is used.
Verification:	Positive (psilocin – red with Solution A, blue with solution B), negative (blank spot plate), record control lot number and results in case notes.
Storage Conditions:	None (single day use)
Expiration:	1 day

Duquenois-Levine

Ingredients:	Solution A: Add 2.5 mL of acetaldehyde and 2 g of vanillin to 100 mL of ethanol Solution B: Concentrated HCl Solution C: Dichloromethane
Verification:	Positive (cannabinoids – blue-violet, blue-violet extracts into lower layer), negative (blank spot plate or test tube)
Storage Conditions:	Solution A – Refrigerated Solutions B and C – Room Temperature
Expiration:	1 year

0.5N NaOH (Methanolic Solution)

Ingredients:	10.0 g NaOH in 500 mL of methanol
Verification:	None required
Storage Conditions:	Room Temperature
Expiration:	1 year

Standards, Training Material, and Reference Materials

Definitions and Scopes of Use

Primary Standards

Definition

Compounds whose origin and composition are known and documented. This is typically expressed as: compound name, name of manufacturing organization, lot or batch number, and date received. Primary standards are preferentially purchased from ISO 17034:2016-accredited suppliers. Refer to the Laboratory Operations Manual Purchasing Procedure for details. Primary standards may be a pure (neat) compound, a solution of a pure compound, or a mixture of compounds (e.g., QC Mixture). All primary standards are verified by GC/MS and/or GC/IR prior to use in casework.

NOTE: Quantitative cannabinoid standards used for THC quantitation may be verified with HPLC-DAD instead of GC/MS or another method. Refer to the THC and THCA Quantitation Procedure Manual.

Maintenance

Primary standards are secured within the chemistry laboratory. They are stored in a locked cabinet, refrigerator, or freezer (as appropriate) in the Standards/Chemical Prep Room #2116. Opening the locked cabinets requires sequential key card swipes from two approved individuals. Keys to the refrigerator and freezer are in one of the locked cabinets. Both individuals must remain in the room until tasks are complete, refrigerator/freezer keys are placed back into the cabinet, and the storage locations are locked.

Receipt of standards is logged in the Primary Drug Standards Logbook along with the Certificate of Analysis (CofA; if available) and the lab-generated confirmation data. See Standard and Reference Material Control for the procedure.

Use

Primary standards may be utilized as reference standards in research, development, and validation of methods, training, quality control of critical reagents, retention time comparison, and for quantitative and qualitative analysis of casework. Primary standards may be used in instrumental analysis to generate spectra, which may be used for comparison with case

sample-generated spectra or to build user-defined libraries. Primary standards may be used to prepare Working Standards.

Secondary Standards

Definition

Compounds whose origin and composition are not documented but exist in the Seized Drug discipline as a result of removal from analyzed cases by present or previously employed analysts, from pharmacies, or other sources of a similar nature. All secondary standards are verified by GC/MS and/or GC/IR prior to use in casework.

Maintenance

Secondary standards are secured in the same fashion as primary standards. These standards are logged in the Secondary Drug Standards Logbook along with the lab-generated confirmation data. See Standard and Reference Material Control for the procedure.

Use

Secondary Standards may be utilized for quality control of reagents, research where purity is not crucial, and training. These compounds may be used for direct comparisons with case samples for qualitative analysis once their identity is verified by GC/MS and/or GC/IR and compared to a literature reference, primary standard, or approved library database.

Working Standards

Definition

Dilute solutions of primary standards.

Maintenance

Working standards are secured within the chemistry laboratory. The stock solution of each working standard is stored in the freezer in the Chemistry laboratory Room #2118. Subsequent vials may be filled from the stock solution and are stored in the chemistry laboratory. See Standard and Reference Material Control for the procedure.

Use

See Primary Standards uses.

Training Materials

Definition

Training materials are substances that are derived from expired primary standards, acquired from police agencies, or other sources of similar nature. Training materials are sufficiently verified to meet the needs of training.

Maintenance

These compounds are secured in the same fashion as primary standards.

A log of all training materials is kept in the Training Material Logbook along with information on the source of the materials and any confirmation data. For more information on receipt and tracking procedures for training materials see Training Material Control.

Use

Training materials are used for training, competency/proficiency tests and displays/demonstrations only.

Laboratory-Established Reference Material (LERM)

Definition

These are materials whose identity and/or composition is determined through third-party laboratory testing. An example of this process is obtaining quantitative THC testing of a plant material sample to establish this sample as a matrix-matched reference material.

Maintenance

LERMs are secured in the same fashion as primary standards.

A log of all LERMs is kept in the LERM Logbook along with the third-party laboratory testing report. See Standard Control for the procedure.

Use

LERMs may be utilized as reference materials in research, development, and validation of methods, training, and matrix-matched quality control samples in casework when purchased primary standards are not available. When purchased primary standards are available, this material is preferable to Laboratory-Established Reference Material.

Standard and Reference Material Control

Traceability

Standards and reference materials will be tracked using a laboratory designated unique identifier. Standard identifiers will include a Control Number and a two-letter designation. Examples: 1-AE, 45-AA, 102-BR. Laboratory-established reference materials (LERM) will also include a Control Number with a two-letter designation with a further suffix of -LERM. Example: 125-AC-LERM or 247-LERM, depending on whether the source material had a two-letter designation at the time of conversion into a LERM.

Any container that contains any amount of substance removed from a primary standard, secondary standard, or LERM will be labeled with the laboratory designated unique identifier.

The list of Control Numbers is maintained by the Standards Maintenance Officer. It is located on the laboratory's SharePoint document library:

Seized Drug Control Number Lists

When new compounds are obtained by the laboratory, the list of Control Numbers will be updated.

Compounds will not change Control Numbers once assigned by the laboratory.

Each new container of a compound will be assigned a two-letter designation that will follow, in alphabetical order, that of the most recent container on hand. Example: A standard container of heroin designated AA is consumed. A new container of heroin is received and designated AB.

The first time a compound is received at the laboratory, the first container will be designated AA.

If more than one container of a compound is received at one time, each container will be given a different two letter designation in alphabetical order. Example: A standard container of heroin designated AA is consumed. Three new containers of heroin are received and designated AB, AC, and AD.

Refer to the laboratory's Health and Safety Manual for chemical receiving procedures when receiving a drug standard.

Primary Standards

Receipt of a New Primary Standard

Compare the standard container(s) to the received paperwork (e.g. packing slip) to ensure the correct product(s) has been received. If any standard container does not match what was ordered, contact the standard manufacturer as soon as possible.

Initial and date the original standard container(s) upon receipt. If multiple containers of the same compound are received, designate each container with the appropriate two-letter designation.

Complete the Drug Standard Control Form including an initial weight (weight for solid standards only (see Standards, Training Material, and Reference Materials). Determine the standard expiration date from the manufacturer information or laboratory policy and record this information on the control form also.

Place the original standard container(s) in the appropriate box (alphabetically according to the common compound name) in the locked cabinet, refrigerator, or freezer depending on the specific storage conditions. Example: The common name for diacetylmorphine is heroin; this drug standard will be in the "H" box.

Add the completed Drug Standard Control Form and a copy of the packing slip to the Primary Drug Standard Logbook in alphabetical order according to the common compound name as in the above example.

Quality Assurance

If available, attach the Certificate of Analysis, or equivalent, to the Drug Standard Control Form.

The standard must be confirmed by GC/MS. The hard copy will be labeled with the respective laboratory-designated unique identifier and the initials of the verifying analyst.

Attach the TIC/Mass spectrum or IR spectrum to the respective Drug Standard Control Form. Confirmation is not required immediately upon receipt of the standard; however, it must be performed prior to the first use of the standard for casework. Only one container per lot number needs to be confirmed. Only one container per lot number should be open at one time.

Portion Control

Primary Standards will be kept under the original manufacturer's seal until opened to portion a sample for testing purposes. The standard will be checked in with an initial weight that includes seals.

All primary standards, except DEA Exempt standards, will be weighed on a calibrated analytical balance both before and after they are portioned for testing purposes. The weight will be recorded on the Drug Standard Control Form. This process will be witnessed by a second person within the Forensic Chemistry Discipline. A top loader balance may be used for standards too heavy or large to be weighed on the analytical balance.

Gross weights will be recorded. The gross weight will include the container with labels, lid, and contents. The initial weight taken when a container is being verified will be done after the manufacturer's seals are removed, to provide uniformity for subsequent sampling weights.

Depletion/Expiration of Primary Standards

When a primary standard has been depleted or expires, the corresponding Drug Standard Control Form will be retrieved, initialed, and dated to record when this occurred. The container itself (including remaining material) may be discarded once the control form reflects that the standard has been removed from service.

The Drug Standard Control Form, with all attachments, will be removed and placed in the Archived Drug Standard Logbook.

If there are multiple original containers of the same primary standard, the Drug Standard Control Form will not be archived until all containers listed on the form are disposed of.

Secondary Standards

Receipt of a New Secondary Standard

Initial and date the original standard container(s) upon receipt. If multiple containers of the same compound are received, designate each container with the appropriate two-letter designation.

Complete the Drug Standard Control Form including an initial weight (weight for solid standards only, see Portion Control). Determine the standard expiration date from the manufacturer information or laboratory policy and record this information on the control form also.

Place the original standard container(s) in the appropriate box (alphabetically according to the common compound name) in the locked cabinet, refrigerator, or freezer depending on the specific storage conditions.

Add the completed Drug Standard Control Form and a copy of the packing slip to the Secondary Drug Standard Logbook in alphabetical order according to the common compound name as in the above example.

Portion Control

All secondary standards will be weighed on a calibrated analytical balance both before and after they are portioned for testing purposes. The weight will be recorded on the Drug Standard Control Form. This process will be witnessed by a second person within the Forensic Chemistry Discipline. A top loader balance may be used for standards too large to weigh on the analytical balance.

Gross weights will be recorded. The gross weight will include the container with labels, cap, and contents. The initial weight will be taken when the container is acquired/created.

Depletion of Secondary Standards

When a secondary standard has been depleted, the corresponding Drug Standard Control Form will be retrieved, initialed, and dated to record when this occurred. The container itself (including remaining material) may be discarded once the control form reflects that the standard has been removed from service.

The Drug Standard Control Form, with all attachments, will be removed and placed in the Archived Drug Standard Logbook.

If there are multiple original containers of the same secondary standard, the Drug Standard Control Form will not be archived until all containers listed on the form are depleted.

Working Standards

Creation of a New Working Standard

Working standards are prepared as needed.

Prepare an appropriate size vial with the following information: compound name, control number, two letter designation of the original standard

container, preparation date (MM/DD/YY), expiration date (MM/DD/YY), and initials of the preparer.

Working standards expire at the same time as the corresponding primary standard. This information may be found on the corresponding Drug Standard Control Form. If a working standard contains more than one component, the expiration date must be no later than the earliest expiration date of the components. Example: If a working standard has 2 components, one of which expires in 8 months and the other in 1 year, the working standard must also expire in 8 months.

Prepare a standard solution (~ 1 mg/mL) using an appropriate solvent. Working standard solutions are not quantitative.

Quality Assurance

Working standards must be able to be traced back to the original standard container(s) they were prepared from.

Portion Control

Approved personnel may portion from a working standard as needed. No weight or witness is necessary.

Depletion of Working Standard

When a working standard has been depleted, the vial will be discarded.

Laboratory-Established Reference Material (LERM)

Receipt of a New LERM

When it is determined that an LERM is needed to support testing, determine the appropriate matrix-matched material needed. Material may be obtained through transfer from law enforcement or purchased from an appropriate supplier. Materials considered expired by the manufacturer are not valid to be converted to a LERM.

The material is accepted and tracked as a Training Material using the existing procedure and controlled forms until such a time as third-party testing establishes it as a LERM.

Determine the critical testing parameters and appropriate testing facility to establish one or more critical attributes of the sample. Examples include but are not limited to:

- Identities of the components of an unknown substance
- Quantitative values such as %w/w THC, THCA, CBD, CBDA, moisture content, etc.

Preference is given to ISO 17025-accredited testing laboratories and the laboratory selection process will verify that the laboratory scope of testing includes the critical attributes of the LERM.

Factors to consider when selecting third party testing facilities should include:

- Accreditation status (preference is given to ISO 17025-accredited testing laboratories)
- The laboratory scope of testing covers the critical attributes to be established and they are reported in an acceptable format (e.g. the reported units)
- Required sample submission quantity and testing methods
- Testing cost and expected turnaround time

Documentation of relevant factors considered should be included on the Vendor Approval Form for the third-party laboratory.

When testing is completed, obtain and review the testing report. If the report is satisfactory, proceed to assigning an LERM control number and completing the LERM Control Form. If the report is not satisfactory for any reason, consult with the Chemistry Supervisor or Technical Lead and the third-party laboratory to determine the next course of action.

Quality Assurance

Attach the third-party testing laboratory report to the LERM Control Form. No additional laboratory confirmation is required once the testing report is determined to be satisfactory.

Portion Control

All LERMs will be weighed on a calibrated analytical balance both before and after they are portioned for testing purposes. The weight will be recorded on the LERM Control Form. This process will be witnessed by a second person within the Forensic Chemistry Discipline. A top loader balance may be used for LERMs too large to weigh on the analytical balance.

Gross weights will be recorded. The gross weight will include the container with labels, cap, and contents. The initial weight will be taken when the container is acquired/created.

Depletion of LERMs

When a LERM has been depleted, the corresponding LERM Control Form will be retrieved, initialed, and dated to record when this occurred. The container itself may be discarded once the control form reflects that the LERM has been removed from service.

The LERM Control Form, with all attachments, will be removed and placed in the Archived Drug Standard Logbook.

If there are multiple original containers of the same LERM, the LERM Control Form will not be archived until all containers listed on the form are depleted.

Drug Standard or Reference Material Breakdown/Degradation

Scope

These requirements apply to primary, secondary, and working standards, as well as laboratory-established reference materials.

Identifying Breakdown/Degradation

Discipline staff may identify when a standard produces an unexpected result in casework. Results associated with the use of this standard may still be used for identification of casework samples when technically justified by the analyst and agreed with the technical reviewer.

If needed, consult the Standards Maintenance Officer to determine disposal requirements.

Impact of Breakdown/Degradation on Expiration Dates

Breakdown/degradation supersedes any manufacturer or laboratory-applied expiration date.

Drug Standard Expiration and Retest Dates

Scope

These requirements apply to primary standards and working standards.

Expiration and Retest Dates

Some drug standards have expiration or retest dates provided by the manufacturer. If these dates are present, they supersede any other expiration dates listed in this manual.

An expiration date generally means that the manufacturer recommends that the standard be disposed after this date. A retest date generally means that the manufacturer recommends retesting the material after this date or determine if it still meets quality requirements. In some cases, the manufacturer will perform this testing themselves. In other cases, the retest date represents a recommendation to the purchaser to perform this testing themselves. In the case of manufacturer retesting, the manufacturer may extend the expiration or retest date of the standard if quality requirements are met.

If no manufacturer expiration or retest dates are provided, an expiration date will be assigned by the laboratory according to the following guide:

Liquid standards: 5 years from the date received

Solid standards: 10 years from the date received

A working standard expires at the same time as the corresponding primary standard. If a working standard contains more than one component, the expiration date must be no later than the earliest expiration date of the components.

Primary standards expire on their manufacturer expiration or retest date unless a new expiration date has been assigned by the manufacturer. If a primary standard has a laboratory-assigned expiration date, it expires on that date.

Training Material Control

Acquisition

Training materials can be acquired from expired primary standards, over-the-counter purchases, or police agencies. The discipline supervisor must approve acquisition of new training materials. Written documentation of this approval must be attached to the Training Material Acquisition Form.

Training materials will have a completed Training Material Acquisition Form that documents all items being received, signatures of the two chemistry

staff members receiving the materials, and signatures of two representatives from the source providing the material.

Completed Training Material Acquisition Form and approvals are scanned and stored in a folder on the Laboratory's SharePoint document library.

Training Material Control Numbers

Training Materials will be assigned a unique laboratory identifier for tracking purposes. The format for the unique identifier is PREFIX-Sequential Identifier.

The prefix is assigned based on the primary drug component in the sample or, in the case of non-controlled mimics, the class will be assigned based on the drug being mimicked. Since the purpose of the training materials is primarily for use with the Seized Drug Training Program, substances that fit within multiple drug categories should be assigned based on how the drugs are classified in the Training Program. Prefixes used by the laboratory are:

Prefix	**Category**
AN	Anesthetics
DEP	Depressants
HAL	Hallucinogen
MJ	Marijuana
NCS	Non-Controlled Substances
NPS	New Psychoactive Substance
OP	Opiates
STR	Steroids
STIM	Stimulants

Example: When a second packet of anesthetic material such as cocaine is added to the inventory, the laboratory identifier would be AN-002 (AN for anesthetics and 002 for the second anesthetics material in the inventory).

The list of Training Material control numbers is maintained by the Standards Maintenance Officer. It is located on the laboratory's SharePoint document library.

Seized Drug Control Number Lists

Each packet of Training Material will be labeled with its training material control number.

Portion Control

When Training Materials are received at the laboratory, a Training Material Control Form will be filled out for each packet of new material. If multiple small bags of the same material from the same source are received, these may be combined upon receipt to make one packet of material.

The Training Material Control Form includes the training material control number, date of receipt, an initial gross weight on a calibrated analytical balance (count may be used for tablets or other items if appropriate), identifying information about the material, and the initials of the two analysts checking in the material.

The Training Material Control Form is also used to document portioning of training materials for use. Prior to taking a sample, the analyst will take an initial weight (or count if appropriate) of the package on a calibrated analytical balance. (A top loading balance can be used if packets are too large or heavy to be weighed on the analytical balance.) The portion is removed, and a final gross weight is recorded along with the reason the portion was removed. The entire process must be witnessed by a second person who initials alongside the preparer. The Training Material Control Form is kept in the Training Material Logbook.

Gross weights are recorded. The gross weight will include the container with labels, lid, and contents.

All training materials are audited annually, and a copy of the audit provided to the discipline supervisor.

Quality Assurance

Any information about the origin and composition of the material should be kept with the Training Material Control Form.

Since training materials can be used for several different purposes, it is up to the user to determine what testing is necessary for confirmation of composition for their intended use. Any confirmatory testing should be included in the Training Material Logbook with the corresponding Training Material Control Form.

Depletion of Training Materials

When a training material has been depleted or is too degraded to continue using, the corresponding Training Material Control Form will be retrieved, initialed, and dated to record when the package was depleted or disposed.

The completed Training Material Control Form, with all its attachments, will be removed from the current Training Material Logbook and placed in the Archived Training Material Logbook.

Logbooks and Contents

The Drug Standard Logbooks are to be stored in the chemistry laboratory. Each respective logbook will contain the following:

- A Drug Standard Control Form for each Primary and Secondary Standard in the inventory.
- A Laboratory-Established Reference Material Control Form for each LERM
- The quality assurance or third-party testing data for each Primary, Secondary Standard, and LERM in the inventory attached to its respective control form and a Certificate of Analysis, if available.
- For Primary and Secondary Standards, a copy of the packing slip from when the drugs standard was received.

The Archived Drug Standard Logbook is to be stored in the chemistry laboratory. It will contain a Drug Standard Control Form, including attachments, for each standard (primary or secondary) that has been depleted, expired, or removed from the inventory but is not yet scanned into the electronic archive.

Electronic Archive

Folders containing scans of archived drug standard and training material control packets are located on the laboratory's SharePoint document library.

At the completion of the annual standard and training material audit (see Drug Standard Security), items in the Archived Drug Standard and Training Material Logbooks will be scanned individually and transferred to the electronic archive.

Drug Standard Security

DEA Registration

The Alaska Scientific Crime Detection Laboratory will maintain a current DEA license for the purchase of controlled substances. A copy of the current DEA license will be kept by the discipline supervisor. The discipline supervisor will

also maintain the laboratory records for purchases of federal Schedule I and II substances (in compliance with DEA Form-222). The laboratory Chief is responsible for assuring that drug standards maintained on the premises are secure.

Drug Standard and Training Material Access

Access to primary standards, secondary standards, and training materials is restricted to the Forensic Chemistry Discipline members, Laboratory Chief and Assistant Chief, Quality Assurance Manager, and critical building maintenance staff. An official memo is submitted at least annually, but updated as appropriate, to the Quality Assurance Manager and the Forensic Laboratory Chief. This memo will list by name the individuals with access to the inventory. A document set containing these access memos is located on the laboratory's SharePoint document library.

If storage of any primary standards, secondary standards, or training materials is to deviate from the previous mentioned locations, this must be approved by the discipline supervisor.

Locked standard and training material locations will be accessed only by approved personnel and only in the presence of a witness.

Drug Standard and Training Material Audit

An audit of all primary standards, secondary standards, working standards, and training materials will occur annually, no later than September 1st.

The audit will account for all primary standards, secondary standards, working standards, and training materials within the laboratory. Inventory of suspected controlled substances received as evidence is not required per 21 CFR § 1304.11 (e) (5). The audit will include, at minimum, the following information for each substance per 21 CFR § 1304.11 (e) (1) (iii) and (iv):

For each controlled substance ready for use in casework or training:

- The name of the substance;
- The drug standard or training material control number
- The lot number (drug standards only)
- The manufacturer name (drug standards only)
- Whether the material is expired (drug standards only)
- The gross weight (solids, liquids not in ampules) or count (ampules, tablets, etc.) recorded during the previous audit
- The gross weight (solids, liquids not in ampules) or count (ampules, tablets, etc.) recorded during the current audit

- Note: Working standards are not weighed

For each substance not available for casework and/or training due to breakdown/degradation or expiration, the audit will include the following (in addition to the above requirements):

- The intended disposition of the material (e.g., disposal, conversion to training material, etc.)

Any expired primary and/or working standards will be removed from the inventory during the annual audit (refer to Disposal of DEA-Controlled Materials). Before removal, it should be verified that the primary standard's expiration date wasn't extended by the manufacturer. If the date was extended, the standard's updated certificate of analysis will be placed in the drug standard control packet instead of removing the standard from the inventory. Secondary standards and training materials have no expiration requirements.

Upon completion of the audit, an official memo will be written by the discipline supervisor to the Quality Assurance Manager and the Forensic Laboratory Chief indicating that this audit was completed. Folders containing annual audit memos are located in on the laboratory's SharePoint document library:

Seized Drugs Standard Audit Records

Disposal of DEA-Controlled Materials

Materials purchased with a DEA license fall into 2 categories – Exempt and Non-Exempt.

Exempt materials are addressed in 21 CFR § 1308.23 and 1308.24 and are not subject to specific disposal requirements. Exempt materials may be washed or rinsed into an appropriate solvent waste container.

Non-Exempt materials (e.g., solid drug standards purchased with a DEA 222 Form) must be disposed of according to 21 CFR § 1317.90 and § 1317.95. On-site destruction is appropriate if using a method capable of rendering all controlled substances to a non-retrievable state (example: RX Destroyer® used according to manufacturer instructions).

Disposal will be documented using DEA Form 41 in addition to the Drug Standard Control Form. Completed DEA Form 41 records are maintained by the Chemistry Supervisor for at least 2 years from the date of destruction.

Archiving Drug Standard and Training Material Forms

After the drug standard and training material audit has been completed, all finalized paper forms will be digitized and store in the laboratory's SharePoint document library. The following types of forms are applicable:

- Training Material Control Forms
- Drug Standard Control Forms
- Training Material Acquisition Forms (usually done at time of acquisition)
- Material Destruction Form (usually done during the drug standard and training material audit)

Training Material Control Forms and Drug Standard Control Forms are located within the binders in the reagent preparation room.

Presumptive Tests

Color Tests

Color tests (also known as spot tests) are non-specific and presumptive in nature. The following color tests are commonly used:

Marquis Test

Place Marquis Reagent in a clean, white spot plate or new test tube along with the sample. Observe and record any color change.

Common Responses:

Violet/Purple	Heroin, Guaifenesin, Buprenorphine
Gray to Violet-black	MDA & MDMA
Orange to Brown	Amphetamine/Methamphetamine & Phentermine
Slow Pink to Rose	Aspirin
Yellow	3,4-methylenedioxymethcathinone & Diphenhydramine
Purple	2,3-methylenedioxymethcathinone

Weber Test

Add a mushroom sample (or alcohol extract) to a clean spot plate. Analyze a psilocyn standard or a known psilocyn-containing mushroom fragment in a separate well. Record the drug standard control number and expiration date in the notes. Add Weber Solution A and look for a red color. Then add one drop of Weber Solution B. A blue color indicates psilocyn. The solution will not turn blue with psilocybin or bufotenine.

Duquenois-Levine Test

Add 1 volume of Duquenois-Levine Solution A to the sample and shake. Then add 1 volume of Solution B. Agitate and observe color produced. A blue-violet color will develop with cannabinoids.

If no blue-violet color is observed, there is no need to continue with the final step.

Add Solution C and note whether the blue-violet color is extracted into the bottom layer. If positive for cannabinoids, a blue-violet color will be extracted into the lower layer.

Physical Identification of Pharmaceuticals

Pharmaceutical preparations may be presumptively identified using a literature reference based on dosage unit, form, shape, color, and/or manufacturer's markings/imprints. Literature reference of pharmaceuticals is appropriate when:

1. Physical identification is done as a secondary test to chemical analysis when an absolute identification is required, or

2. When a pharmaceutical preparation is not selected for analysis to document the reason the item was triaged.

The following are accepted references for the physical identification of pharmaceuticals:

- Physician's Desk Reference (PDR)™, Medical Economics
- Drug Identification Bible™, Amera-Chem, Inc.
- Manufacturer sealed and marked products (e.g., blister packs, sublingual film packaging, etc.)
- Online resources such as government and manufacturer websites, Pharmer.org, Drugs.com, RxList.com
    - Printed documentation of the reference must be included in the case notes when using a website as a reference. For example, a PDF printout or screenshot of the webpage. The website URL must be noted.

Botanical Identification of Plant Material (Cannabis Only)

Observe sample under stereomicroscope.

The minimum criteria for a positive microscopic examination of suspected Cannabis are cystolithic hairs and clothing hairs on opposite sides of the same leaf.

Thermo Scientific "TruNarc" Analyzer

The TruNarc analyzer is a handheld 785-nm Raman spectrometer. The Seized Drug discipline uses it for presumptive analysis of suspected drug substances.

Maintenance

NOTE: Do not leave TruNarcs plugged in longer than necessary for charging as this damages the battery over time.

There are no user-serviceable components inside the TruNarc analyzer. All service operations (e.g., to address freezing or not turning on) must be performed by Thermo Fisher Scientific or an authorized service agent of Thermo Fisher Scientific.

Periodically, the TruNarc analyzer should be cleared of test records and updated using the associated computer software. Test results are contemporaneously recorded into each case for which it is used; therefore, the clearing of test records does not impact the validity of testing.

Quality Assurance

The Self Check is a diagnostic test that verifies that the analyzer is operating according to its factory settings. On the first working day of the week, a Self-Check must be performed (with a passing result) before the TruNarc can be used for casework.

Note: A Self Check at the start and end of every work shift, as recommended in the manufacturer's user guide, is not required. This is because the instrument is only being used for presumptive analyses that are performed in a laboratory setting.

During the Self Check, the TruNarc analyzer scans the Self Check standard (polystyrene) that is attached to the nose cone.

Results of all Self Checks will be documented on the TruNarc Self Check Form. If a TruNarc repeatedly gives a failed result, it will be taken out of service until the issue can be resolved. The TruNarc Self Check form will be periodically scanned and archived on the laboratory's SharePoint document library (Digitally Archiving Instrument Records).

Analysis

Procedure for TruNarc analysis:

- Turn on the TruNarc analyzer and activate the laser. Select **Scan** in the Welcome screen.
- Select **Scan** in the Scan Ready Screen. The scan will begin immediately. The laser light glows when the laser is active.
- When the scan finishes, the laser indicator light turns off, and the instrument begins analyzing the data. The sample can now be moved away from the analyzer. When the analysis step finishes, the analyzer displays a result screen.

Record which TruNarc was used, any sample preparation, and the displayed result in the case notes. If the operator aborts the scan prior to the 90 second timeout feature, the result will be recorded as "Aborted".

Extraction Protocols

Many drug samples are mixtures or contain excipient material requiring the compound of interest to be separated from a matrix before subjecting the sample to further analysis. Information on solubility and physical properties can be found in *Clarke's Isolation and Identification of Drugs and Poisons* or other appropriate references. The choice of an organic solvent is dependent upon the drug to be extracted and the preference of the analyst.

GC/MS injection solvents used for case samples and their respective negative controls will contain an internal standard (typically tetradecane at ~0.5 mg/mL).

Residue Collection

- Paraphernalia with visible residue may be:
    - Swabbed with a cotton-tipped applicator and treated with an extraction listed below
    - Scraped and extracted appropriately
- If the swab method is chosen, a negative control must be prepared using a clean swab at the same time.

General Extractions

Dry Extraction

- Place a portion of the sample in a disposable test tube
- Add a solvent that dissolves the drug of interest
- Vortex or shake
- If necessary, decant or filter to separate the solvent and discard any insoluble material

Liquid/Liquid Extraction

- Basic extraction
    - Place a portion of the sample in a disposable test tube
    - Dissolve the sample in borate buffer (or other suitable basic solution)
    - Add appropriate solvent
    - Vortex or shake
- Acid extraction
    - Place a portion of the sample in a disposable test tube
    - Dissolve the sample in suitable acid
    - Add appropriate solvent
    - Vortex or shake

- Acid/base extractions (back extractions)
  - Place a portion of the sample in a disposable test tube
  - Dissolve sample as described in the acid extraction above
  - Add an appropriate organic solvent without internal standard
  - Vortex or shake to separate layers and discard organic layer
  - Adjust pH of acidic aqueous layer to make basic
  - Add appropriate solvent
  - Vortex or shake

Food Products with Suspected THC (for qualitative identification)

- Place a portion of sample in a disposable test tube
- Add hexanes
- Vortex or centrifuge
- Transfer hexanes to a new disposable test tube
- Extract with 0.5N NaOH (methanolic solution)
- Resulting layers:
  - Top hexanes layer – Discard
  - Basic methanolic layer – THC, if present
- Wash basic methanolic layer with three aliquots of hexanes
- Acidify using 1N HCl to pH 1-2
- Extract with hexanes
- Vortex or shake
- Resulting layers:
  - Top hexanes layer – THC, if present
  - Acidic methanolic layer – Discard
- Dry hexanes layer to completeness and reconstitute in an appropriate solvent

Gamma-butyrolactone (GBL) or 1,4-Butanediol (BD) Extraction

- Combine approximately equal volumes of the sample liquid and dichloromethane with internal standard in a test tube (2-3 mL of each, when sample size permits). If the sample is powder, dissolve in water and extract with dichloromethane
- Vortex or shake
- Let settle
- Resulting layers:
  - GBL/BD will be in the dichloromethane layer
  - Gamma-hydroxybutyric acid (GHB) in the aqueous layer, if present

Alkaloid Extraction

- Grind sample to a fine powder
- Soak 1 to 2 grams in ethanol or methanol for 30 minutes
- Vortex
- Add 10 drops of 20% acetic acid
- Soak an additional 30 minutes
- Vortex
- Centrifuge and transfer acidic ethanol to a test tube
- Evaporate to dryness
- Reconstitute with dichloromethane
- Vortex
- Analyze by GC/MS

Mushroom Extraction

- Warm up evaporator
- Finely chop ~0.3 grams of sample and transfer to a large test tube
- Powder sample with dowels, if desired (very weak/negative Weber)
- Add 1-2 mL of deionized water (Also make a blank tube at this point)
- Add 20% acetic acid to the suspended sample until a pH of about 3-4 is reached
- Wash sample 3 times with dichloromethane (no internal standard), centrifuging and disposing of the dichloromethane layer (lower layer) each time
- Basify sample with ammonium hydroxide to about pH 9
- Extract sample in dichloromethane (no internal standard) and transfer dichloromethane to a smaller test tube
- Evaporate sample and blank to dryness
- Reconstitute in DCM with internal standard and transfer to a vial with an insert.

Mushroom Chocolate Extraction

- Warm up evaporator
- Finely chop ~1 chocolate square and transfer about half of the chopped portion to a large test tube.
  - Reserve the other chopped portion for Weber test
  - Weber test may require Mushroom Extraction, due to chocolate interference with the Weber)
- Add 1-2 mL of deionized water (Also make a blank tube at this point)
- Add 20% acetic acid to the suspended sample until a pH of about 3-4 is reached
- Optional: Centrifuge and decant the liquid to remove solids

- Wash sample 3 times with dichloromethane (no internal standard), centrifuging and disposing of the dichloromethane layer (lower layer) each time
- Wash the aqueous portion 3 times with hexane, centrifuging and disposing of the hexane layer (upper layer) each time
- Basify sample with ammonium hydroxide to about pH 9
- Extract sample in dichloromethane (no internal standard) and transfer liquid to a smaller test tube
- Evaporate sample and blank to dryness
- Reconstitute in DCM with internal standard and transfer to a vial with an insert for injection.
- Analyze by GC/MS

Derivatization of GHB

- Refer to: Derivatization of GHB Validation
- Place the test sample or standard (or extraction thereof) in a glass vial or test tube. *NOTE: GHB does not extract well into Borate Buffer/CH2Cl2
- Heat at about 55°C for about 30 min in the oven.
- Add approximately 100 uL dichloromethane with internal standard, vortex.
- Add approximately 100 uL BSTFA with 1% TMCS, vortex.
- Transfer contents to a capped GC-MS sample vial.
- Allow sample to incubate at room temperature for about 15 minutes.
- Analyze on GC-MS.

Reporting Conclusions of Testing

General Reporting Requirements

To clearly state on reports what analysis was performed, items tested will be sub-itemized when items of the same population were not tested.

Approved report language is listed below. Alternative wording on reports is not permitted without prior approval from the Technical Lead or discipline supervisor. Approval must be documented in the case file.

When one or more controlled substances are identified, the report will state the name of each controlled substance. Reported names of controlled substances will match those listed in the Alaska Statues, where possible.

When the presence of a non-controlled substance(s) affects the schedule of a controlled substance, the report will also identify the non-controlled substance.

When cocaine is identified and a TruNarc analyzer result lists the base or hydrochloride form of cocaine, the report will state Cocaine followed by the corresponding form.

When a controlled substance is not detected, even if non-controlled substances are identified, the report will state:

"**No controlled substances per Alaska Statutes detected**."

After determining that no controlled substances are present, an identified non-controlled substance may be reported.

When a small sample is present and there is not enough for complete testing while maintaining a portion untested, the report will state:

"**Quantity insufficient for analysis unless written approval to consume the evidence is provided by the District Attorney's Office**."

When a small sample is present and there is not enough to meet the requirements of the Analytical Scheme, the report will state:

"**Quantity insufficient for analysis.**"

When testing has begun on a sample but not enough exists for complete identification the report will state:

"**Insufficient sample for identification unless written approval to consume the evidence is provided by the District Attorney's Office**."

When, in the analyst's opinion, the apparent purity of the only indicated controlled substance is too low to warrant pursuit of identification, the report will state:

"**Sample consistency precludes identification.**"

Items that were not analyzed or only a physical identification was performed for triaging purposes will be reported as:

"**Not Analyzed.**"

Qualitative Reporting of Suspected Marijuana or THC Products

NOTE: The approved language from this section may be copied from this manual into the "Additional Comments for Report (Item Level) field on the Reporting tab in the Seized Drugs custom forms in JusticeTrax.

When reporting *Cannabis* or a cannabinoid-containing product for qualitative analysis, the item description, item weight, and identified substances are reported in the same manner as other cases. The reporting language will additionally include a statement that quantitation is required to determine the schedule an indication to the customer that action is needed to initiate this process.

Example: "Cannabis" *and* "Tetrahydrocannabinol" *and*

> "**Quantitative analysis is required to determine the controlled status of this item. Please contact the lab for further testing options. The District Attorney's office must contact the laboratory if quantitation is required on this sample."**

Note: The minimum quantity of remaining sample for THC quantitation via NMS Labs (Horsham, PA) is 0.085 gram (85 milligrams) of any matrix type.

Reporting Cannabinoids When Other Controlled Drugs Are Identified

The determination to pursue THC quantitation is based on determining the potential controlled status of the item. If any controlled substance (per Alaska Statutes) is identified in addition to delta-9-tetrahydrocannabinol, third party THC quantitation is not recommended for that item.

The names of each identified controlled substance will be reported as specified in General Reporting Requirements.

Administrative

Evidence Control

All evidence transfers are documented in the LIMS. The analyst will ensure all evidence has transferred properly in the LIMS either through the electronic chain of custody or appropriate evidence report.

Personal evidence lockers are provided for each analyst in the Seized Drug laboratory. Additional larger storage areas are available in the Seized Drug evidence room # 2115. Each analyst will ensure all evidence is properly stored prior to leaving the laboratory. After opening, evidence items will be secured in a laboratory provided package before returning. Any deviations from this practice will be documented in the notes.

If an analyst observes that the outermost seals are not intact on an evidence package containing controlled substances or currency, the analyst shall document the condition of the package in LIMS. Additionally, a witness shall attest to the condition of the package in LIMS. Refer to the Evidence Room Manual for more information.

Evidence Handling and Marking

Each time an item is opened, the outer packaging will be marked with, at minimum, the analyst initials and date the item was opened. Inner layers of sealed packaging do not require initials and date when opened.

Analysts shall ensure all items can be identified throughout the testing process. Each item analyzed will be marked with the laboratory case number and item number (or subitem number if relevant). If it is not possible to mark the item itself, the innermost layer of packaging will be labeled, or the item can be repackaged into marked laboratory packaging. Items that require further testing on the packaging (e.g., Latent Prints) shall be repackaged rather than marking the evidence directly. All laboratory packaging added to evidence items shall be marked with "repackaged" or similar wording to clearly distinguish it from potential evidence items.

Subitems shall be created when items contain multiple testable components. A subitem will be created for each testable component with an individual result.

Example of an acceptable approach: Item 1 contains 5 tablets, and one tablet is analyzed. Subitem 1-A contains the testing record for the tablet analyzed. Subitem 1-B represents the remaining unanalyzed tablets with a reported result of Not Analyzed.

In instances where an item contains components that are clearly not testable (e.g., a purse is submitted with various contents in addition to testable materials), the untestable components can either 1. Be described in the parent item with the testable components sub-itemized, or 2. Have a separate subitem created for the non-testable items with a result of Not Analyzed.

When analysts remove a portion of an evidence item for testing, the analyst shall mark final autosampler vials with enough of the laboratory case number and item number to clearly maintain the identity of the sample throughout processing. Analysts are not required to mark test tubes used for extraction or temporary storage of sample if the sample is immediately extracted and placed in an autosampler vial. Test tubes that are not labeled with laboratory number and item number may not be utilized after the initial transfer. In this instance, if additional sample is needed, the analyst shall return to the original test item.

If evidence is reopened later, this information will be documented in the notes and a second seal applied.

Evidence Discrepancies

Significant discrepancies between the agency item description and the actual evidence contents will be noted.

Some types of discrepancies require further documentation. The analyst is responsible for determining when one (or more) of these discrepancies has occurred. Key examples and procedures for documentation are listed below:

1. When less than the amount of material described in the agency item description (e.g., fewer tablets, lower weight) is present…
    a. Item contents must be witnessed by another laboratory member. The analyst will describe the discrepancy and record the witness's name in the case notes. The person witnessing the discrepancy will document their observations in an email to the discipline supervisor and the case analyst. The case analyst will place a copy of the email in case activities under SD-Evidence Discrepancy. The technical reviewer is responsible for ensuring the email from the witness is present in the case file, was added by the analyst, and adequately describes the discrepancy.
2. When the items contents differ in appearance from the agency item description (e.g. contents are white powder but dark substance is listed on the RLS)…
    a. The analyst must contact the submitting agency to clarify the discrepancy. A record of this contact will be placed in case activities

under SD-Evidence Discrepancy by the case analyst. The technical reviewer is responsible for ensuring the record is present in the case file and adequately resolves the discrepancy.

Discrepancies may be addressed by the discipline supervisor, if necessary.

Permission to Consume with a Latent Print Request

If an item has a pending latent print request and the Seized Drug analyst determines that Seized Drugs testing is not possible without consuming the remaining evidence, the analyst will select "Latent Prints (permission to consume)" from reporting language dropdown. The following reporting language will appear on the laboratory report for that item:

"The quantity of suspected controlled substance is insufficient for analysis while leaving a portion untested. This item also has a latent print request. To prevent potential destruction of suspected controlled substance evidence, latent print testing has been postponed for a maximum of 30 days from the report issue date. In order to complete testing for controlled substances, the District Attorney's Office must provide written approval to consume the remaining suspected controlled substance within 30 days. If written permission is not received, no further testing will be performed on this item and it will be returned to the submitting agency."

Batching Instrument Tests

In some cases, it may be beneficial to have one analyst start an instrumental analysis containing sample extracts prepared by a different analyst. In this circumstance, the analyst preparing the sample extract must:

1. Verify that the quality control requirements for the instrument have been met prior to deciding that the instrumental data are acceptable for use AND
2. Ensure that the instrumental data are properly identified including the case number, item number (if applicable), analyst initials, and date

Testing Reports

A report will be issued for each case analyzed. Refer to the Laboratory Operations Manual for specific requirements on the content of laboratory reports. Reporting language used for Seized Drug testing is listed in the Reporting Conclusions of Testing section.

Analysis End Date

The analysis end date is when the report is initially marked Draft Complete. If a case requires additional analytical work, the analyst will add a new end date once

this work is complete and the case is forwarded to technical/administrative review. In this situation, the original end date is placed in the notes by the analyst. If a case requires only administrative changes to the notes or the report, a new end date is not required.

Technical Record Contents

The Seized Drugs technical record encompasses the laboratory report as well as the instrumental data and attached reference, where applicable. The technical record will include, at a minimum:

- The start and end dates of analysis.
- A detailed description of each items' packaging and contents. This must include enough detail to properly convey the information to the technical and administrative reviewer.
- The weight of each item analyzed.
- The sampling plan applied, when appropriate.
- The sample preparations or extractions used.
- Descriptions of each analysis performed, including results.
- Positive and negative control results for color tests, where applicable
- Supporting documents (e.g., instrumental data), where applicable
- For controlled substance not specifically listed in the relevant Statute, documentation of the attributes resulting in the controlled status (see Scope of Testing)
- The conclusions reached for each item (including items that were not analyzed).
- The dates of each Lab Activity (refer to Appendix 1 - Lab Activity Dates)

When items are not selected by the analyst for analysis, based on information the analyst observes on the evidence packaging or within the evidence, the reason must be documented in the notes.

Amendments to Technical Records

For instrument reports and other request-specific attachments in JusticeTrax (such as pictures, spreadsheets, printed webpages, etc.), amendments to these attachments after the request was turned in for technical review must be made in such a way that the original data is retained, the altered aspects are clear, the person responsible for the change is identified and the date on which the change was made is listed.

Examples of this for Seized Drugs include a digital correction to an instrument printout or regenerating a PDF of a spreadsheet for weight calculations.

Additional Testing Reports

A separate report will be issued for additional testing requests that are related to items previously addressed in another Seized Drug report.

Examples of additional testing reports include:

- When items that were previously reported as not analyzed are subsequently analyzed.
- When written approval to consume the evidence is provided by the District Attorney's Office after a report was issued indicating the quantity was insufficient for analysis.

When an additional testing report is issued, it will reference the original report and state the reason for additional testing.

Technical and Administrative Review

The evidence must be returned to the evidence return location or another laboratory member prior to administrative review. Each case will be technically and administratively reviewed prior to distribution. The minimum requirements listed in the Laboratory Operations Manual will be completed.

The Technical Lead or discipline supervisor will be consulted on any issues between the case analyst and the technical reviewer and will make the final decision.

Preliminary results may be provided by the case analyst when necessary. This communication will be documented in the case file and will include a disclaimer that this is preliminary information. The Laboratory Operations Manual outlines further details on the release of preliminary results under 5.10.3.3.

Release of Preliminary Results

In some cases, GC/MS results will indicate the presence of a compound that may be of interest to the case agent, but conclusive identification and reporting of the compound is not pursued (i.e., psychoactive compounds not currently scheduled in Alaska). In these situations, the analyst may communicate preliminary results to the case agent following the procedure outlined in the Laboratory Operations Manual.

Amended Reports

An amended report will be issued when an error is discovered after the report has been distributed. Refer to the Laboratory Operations Manual for required procedures on documenting amended reports. The case analyst will notify the discipline supervisor when an amended report is issued.

Proficiency Tests

The discipline supervisor or designee will destroy the proficiency test samples only after the results have been received from the proficiency test provider.

External proficiency tests are reported based on the Federal Regulations under the United States Controlled Substances Act to comply with the proficiency test provider's guidelines.

Digital Archiving of Paper Records

Paper records generated in the normal course of business will be converted to digital documents at once per calendar year. The following types of records in Seized Drugs are initially paper and require digitization:

- TruNarc Self Check Forms (stored in the TruNarc binders in the lab space)
- Balance Performance Check Forms (stored in the balance binders in the lab space)
- Reagent Check Forms (stored in the reagent binder in the reagent preparation room)
- Reagent Preparation Form (with verification data attached; stored in the reagent binder in the reagent preparation room)

Discovery

Discovery for the Seized Drug discipline is managed by the discipline supervisor other than routine bench note requests. Discovery requests are stored in the LIMS. Refer to the Laboratory Operations Manual for more information.

Quarterly Seized Drugs Retesting Program

Proficient analysts in the Seized Drug discipline will have one request per quarter reworked by another proficient analyst after the completion of the technical review but before the report has been released. The reports generated for both the original analysis and the retesting analysis will be released as the final report for the request. Refer to Seized Drugs Retesting Program Working Instructions for more information.

Each quarter the Seized Drug Technical Lead will provide a report to the discipline supervisor summarizing all retesting results. Copies of these reports are stored on the laboratory's SharePoint document library.

Performance Monitoring Program

The current ANAB Scope of Accreditation to ISO/IEC 17025:2017 lists the Key Equipment and Technologies used within the Seized Drugs discipline for Qualitative Determination (identification) and Weight Measurement of seized drugs.

Performance monitoring of these techniques is explained in the Seized Drugs Retesting Program Working Instructions.

Continuing Education of Seized Drugs Analysts

Seized Drugs analysts are required complete continuing education to stay up-to-date on topics such as emerging drugs, new technologies, instrumentation, and forensic standards of practice. Other terms used to refer to continuing education may include training and professional development.

Each analyst should average 16 hours per year of continuing education over a 3-year cycle. This may be in person with an instructor, distance learning, or computer-based. Additional requirements for continuing education are as follows:

1. Continuing education shall be relevant to the laboratory's mission:
    a. If it is unclear whether a training or continuing education opportunity is relevant, consult with the Technical Lead or discipline supervisor.
2. Continuing education shall be documented in JusticeTrax and must include one or more of the following:
    a. Literature review documentation (see Literature Review Requirements)
    b. Issuance of a certificate or diploma
    c. Publication of a paper
    d. Verification of attendance
    e. Recording of a presentation or exercise
3. Continuing education may be obtained from a variety of sources including, but not limited to:
    a. Chemistry courses taught at the postsecondary educational level (i.e., college or university)
    b. Instrument operation/maintenance courses taught by vendors
    c. On-the-job classes conducted by employers
    d. Webinars from academic or forensic organizations
    e. Current literature review (see Literature Review Requirements)
4. Literature Review Requirements: Literature review may be counted towards continuing education requirements if one of the following criteria are met:
    a. Read an article and participate in a group discussion with other lab staff:
        i. Meeting minutes showing attendance will be stored in the individual training record in JusticeTrax
        ii. Up to one hour of continuing education credit may be claimed for completion
    b. Read an article then write an assessment of the article content:
        i. An assessment may include, but is not limited to:

1. Considering the pros and cons of the information presented
2. Developing an informed opinion as to whether the practice/procedure would be beneficial to the laboratory
3. Identifying limitations or alternatives

ii. The written assessment will be stored in JusticeTrax as documentation of the literature review
iii. Up to one hour of continuing education credit may be claimed for completion

Extended Absence Policy

When an analyst is away from the laboratory for an extended period (six months or longer), he/she will be required to successfully complete an internal competency test before resuming casework analysis. The scope of the competency test and authorization to resume casework are the responsibility of the discipline supervisor.

Instrument Validation Requirements

When new instrumentation is acquired, the manufacturer's representative will install and perform the initial set up of the instrument when needed.

Prior to use in casework, a validation of the performance of the instrument will be performed prior to the instrument being available for use in casework. A validation plan/proposal must be approved by the discipline supervisor or their designee before the validation study commences. The validation plan/proposal will incorporate sufficient elements to demonstrate that the method is fit for intended use.

Completed instrument validation reports are located on the laboratory's SharePoint document library.

Method Validation/Verification Requirements

When a new testing method is being evaluated for use by the discipline, the Technical Lead or discipline supervisor will determine whether a full validation is needed or if a verification is sufficient. Factors such as the complexity of the method and acceptance within the forensic community will be considered in this decision.

Validation or verification of the performance of the method will be performed prior to the method being available for use in casework. A validation or verification plan/proposal must be approved by the discipline supervisor or their designee before the study commences. The plan/proposal will incorporate sufficient elements to demonstrate that the method is fit for intended use.

Completed method validation/verification reports are located on the laboratory's SharePoint document library.

Sampling Plan

Overview

A sampling plan is a statistically-valid approach (see References 1.) for testing a subset of items that may result in an inference as to the identity of untested items. The sampling approach and testing hypothesis must be decided before testing begins. A sampling plan is not used by default on large populations and must be agreed upon by the Chemistry Supervisor and the assigned attorney for the case.

Example: Hypothesis is that all tablets within the item contain fentanyl.

NOTE: Based on the intent of the Analytical Scheme, the hypothesis will be that the tested units contain one or more specific, identifiable controlled substances. The purpose of a sampling plan is NOT to look for the presence of all possible controlled substances, only that/those identified in the hypothesis.

Sampling approach: _____ tablets from the population will be analyzed with the assumption that all will contain fentanyl. The number of tablets chosen for testing is based on total number of tablets to which the inference may be applied (whether counted individually or extrapolated), and a determination of the coverage probability to be used (e.g., 95% confidence that 70% of the units contain fentanyl).

Deviations from the sampling plan require prior approval from the Technical Lead and/or discipline supervisor and will be documented via a Deviation Request Form.

Tracking Sampling Plan Use

When a sampling plan is used, "Sampling Plan" will be selected under the request Complexity in JusticeTrax. This selection allows these cases to be located for auditing purposes.

Determining Populations

The use of a sampling plan requires that all units appear to be similar in appearance. The analyst is responsible for determining visual similarity and may use unit shape, color, dimensions, markings/imprints, correspondence, or some combination of factors to make this determination. The analyst will document the attributes that lead to this determination in the notes if a sampling plan is to be used.

Extrapolation of Count

If the count of units in the population has been estimated by weight extrapolation, the population size will have uncertainty associated with the estimate. For the purpose of determining the appropriate number of units to test, use the population

size (*N*) row in the table that is closest to but not less than the estimated count plus the uncertainty.

Example: Estimated 5,000 +/- 100 tablets, use the population size row for 10,000 units.

Hypergeometric Sampling Plan

The hypergeometric sampling plan is one statistically-valid type of testing approach. The following table (parts 1 and 2) illustrates the appropriate number of units to test for various population sizes (*N*), confidence levels, and proportion of the population (*k*). Note that the values listed in this table assume that all tested units are positive. The UNODC Guidelines on Representative Drug Sampling ST/NAR/38 contains tables for situations in which one or more negative tests are expected.

Population size *N*	95% confidence			99% confidence		
	k=0.5	*k*=0.7	*k*=0.9	*k*=0.5	*k*=0.7	*k*=0.9
10	3	5	8	4	6	9
20	4	6	12	5	9	15
30	4	7	15	6	10	20
40	4	7	18	6	10	23
50	4	8	19	6	11	26
60	4	8	20	6	11	28
70	5	8	21	7	12	30
80	5	8	22	7	12	31
90	5	8	23	7	12	32
100	5	8	23	7	12	33
200	5	9	26	7	13	38

Table 1, Part 1 from UNODC Guidelines on Representative Drug Sampling (see References 1.)

Population size N	95% confidence			99% confidence		
	k=0.5	k=0.7	k=0.9	k=0.5	k=0.7	k=0.9
300	5	9	27	7	13	40
400	5	9	27	7	13	41
500	5	9	28	7	13	41
600	5	9	28	7	13	42
700	5	9	28	7	13	42
800	5	9	28	7	13	42
900	5	9	28	7	13	43
1 000	5	9	28	7	13	43
5 000	5	9	29	7	13	44
10 000	5	9	29	7	13	44

Note: Required sample size to guarantee with 95% or 99% confidence that the seizure contains at least a proportion of k drugs, if it is expected that all sampled units contain drugs.

Table 1, Part 2 from UNODC Guidelines on Representative Drug Sampling (see References 1.)

Population size	k = 0.5, 95% confidence	k = 0.7, 95% confidence	k = 0.9, 95% confidence	k = 0.5, 99% confidence	k = 0.7, 99% confidence	k = 0.9, 99% confidence
65,000	5	9	29	7	13	44

Table 1, Part 3 from the ENFSI DWG Sampling Calculator for a population size of 65,000 (see References 3. and 4.)

Conducting Testing

After the sampling approach has been decided (and approved, if required), testing may begin. The results of each analysis will be recorded in LIMS as normal. If any units test negative and the sampling approach assumed all units would be positive, the sampling plan must be reevaluated. Consult with the Technical Lead or discipline supervisor if this occurs.

Reporting Sampling Plan Results

Reported results for an item (or sub-item) on which a sampling plan was used must clearly identify which drug(s) was/were identified, the total weight of material to

which the inference of identity is being applied, identification of the sampling plan used, the confidence level of the conclusion, and the guaranteed proportion of the population to which the inference is applied.

Uncertainty of Measurement

Uncertainty Background

The estimation of the uncertainty of measurement of seized drug weight determination has been performed for all models of balances in used for casework. For the MX2002 and XSR16001L balances, the initial studies were performed in 2024. For the XSR204 analytical balance (Chemistry Analytical #1, serial number C123900380), the initial study was performed in 2022. Balance uncertainty reports for current models are locate in Seized Drugs Uncertainty of Measurement Reports.

MX2002 and XSR16001L balances are used for routine case while the XSR204 balance is primarily used for THC quantitation sample weighing.

Initial Uncertainty Report Contents

The uncertainty of measurement reports address the following elements:

- Statement of the measurand (sample weight measured in grams)
- Statement of the measurement traceability
- Equipment used
  - Balance serial numbers
  - Reference masses
- All uncertainty components considered
- All significant uncertainty components and their evaluation
- Data used to estimate repeatability and/or reproducibility
- All calculations
- The combined standard uncertainty, coverage factor (k-value), coverage probability, and resulting expanded uncertainty
- The schedule to review and/or recalculate the measurement of uncertainty

Check Standards and Equipment

The check standard chosen for the Mettler Toledo MX2002 balances is tablets packaged for stability. The check standard for the Mettler Toledo XSR16001L balances is a powder packaged for stability.

The check standard for the Mettler Toledo XSR204 balance is a capped 50 mL polypropylene tube mimicking the sample weighing container used for plant material in THC quantitation. Check standard measurements for Chemistry Analytical #1 involve the use of an anti-static device which is used every time prior to the check standard weight measurement.

Combined Weight Uncertainty

The 2013 uncertainty of measurement reports address uncertainty of combined weights utilizing a root sum of squares formula. The reported uncertainty of a combined weight is calculated as follows:

balance uncertainty x the square root of the number of measurements made ($\sqrt{N}$)

where the balance uncertainty is the current expanded combined uncertainty for the balance used at 95% coverage. If the value obtained from calculation above has more decimal places than the readability of the balance utilized, the calculated value will be rounded to the readability of the balance (i.e., rounded up at a 5 or higher and rounded down at a 4 or lower).

Ongoing Estimations of Uncertainty

An ongoing estimation of the uncertainty of measurement is performed utilizing data from weekly and monthly balance checks. The data from this ongoing estimation of the uncertainty of measurement will be reviewed and updated reports will be written approximately every 3 years, or if any significant change in the expanded uncertainty is detected. Each estimation of uncertainty must contain the elements included in Initial Uncertainty Report Contents.

Note: A shorter balance uncertainty review period from a previous uncertainty report supersedes the 3-year requirement (e.g., entirely new models of balance).

The initial reports and updates are stored on the laboratory SharePoint document library.

Expanded Combined Standard Uncertainty

The stated uncertainties below are valid only for balances used at the Alaska Scientific Crime Detection Laboratory.

The current expanded combined standard uncertainty at 95% coverage (k=2) for the Mettler Toledo MX2002 balances is +/- 0.04 gram.

The current expanded combined standard uncertainty at 95% coverage (k=2) for the Mettler Toledo XSR16001L balances is +/- 0.3 gram.

The current expanded combined standard uncertainty at 95% coverage (k=2) for the Mettler Toledo XSR204 balance (S/N: C123900380) is +/- 0.0022 gram.

Seized Drugs Key Control Policy

Each analyst is assigned the evidence locker in their work area. Any unassigned work area evidence locker may be used, as needed. When not in use, the key is stored in the lock indicating it is available for use.

Most of the evidence lockers in the evidence room #2115 are unassigned and available for use, as needed. When not in use the key is stored in the lock indicating it is available for use.

If a locker key is lost, the discipline supervisor must be notified immediately.

The following are the unique key numbers for the evidence lockers in room #2115.

- 3743
- 3748
- 3754
- 3761
- 3779
- 3796

The following are the unique key numbers for the evidence lockers in the work areas.

- 3027
- 3091
- 3059
- 3060
- 3062
- 3063
- 3064
- 3158

References

1. UNODC Guidelines on Representative Drug Sampling 2009.pdf
2. Measurement Uncertainty for Extrapolations of Net Weight and Unit Count Supplemental Document SD-6, SWGDRUG, 2017-07-10 SD-6_MU for Extrapolations_10162017.pdf (swgdrug.org)
3. ENFSI DWG Sampling Calculator Revision July 2017
4. 2023.12.22 Verification of ENFSI DWG Sampling Calculator stored in SharePoint

Appendix 1 - Lab Activity Dates

The following lab activities are conducted during Seized Drugs testing. The analyst records the dates of each lab activity in the technical record. The technical record is composed of the laboratory report as well as the instrumental data attachments. This table lists the recommended location where the lab activity dates are recorded and is intended to be used as a guide for the analyst and technical reviewer. Other locations in the technical record may be used for a lab activity date upon agreement between the analyst and the technical reviewer.

Activity Name	Location of Date
Weight	Worksheet section of report
Marquis	Worksheet section of report
TruNarc	Worksheet section of report
Other Color tests	Worksheet section of report
Physical Identification	Worksheet section of report
Extractions	Worksheet section of report
Botanical ID for Cannabis	Worksheet section of report
GC Retention Time Analysis	Date from instrument printout
GC/MS	Date from instrument printout
GC/IR	Date from instrument printout
Instrument Data Interpretation	Worksheet section of report

NOTE: Some lab activities may have more than one date if they are performed more than once or, for example, instrumental data are interpreted on different days.

Appendix 2 – Targeted GC/MS Methods

Targeted Method Background

Targeted methods may be used in certain situations to achieve analytical goals (e.g., earlier elution, more complete separation) when the standard SCREENGHALF or SCREENGX1 method will not achieve those specific goals.

Targeted Method Requirements

Example standard methods are saved in the laboratory's SharePoint document library (Seized Drugs Instrument Records); however, due to instrument-to-instrument differences in detector parameters, a targeted method shall always be created from the approved SCREENGHALF or SCREENGX1 methods on the same instrument. Because targeted methods may not be updated for changes to gain factor, these methods should be deleted after use.

The method parameters that were used for a case, if different than the approved SCREENGHALF or SCREENGX1 methods, will be documented in the case file.

Note: Some targeted methods will cause the internal standard, tetradecane, used in the injection solvent to elute in the solvent front. This is expected behavior when the starting oven temperature becomes sufficiently high. Refer to GC/MS Acceptability Criteria.

Targeted Method Parameters

Targeted method parameters may include changes to one or more of the following settings:

- Oven temperature program (initial temperature, ramp, hold times)
- Method run time/end time
- Injector temperature
- Injector split ratio

Example Targeted Methods

Fentanyl - Hypergeometric Sampling Plan

Oven: 290 Celsius, isothermal (no ramp)
Run Time: 7 minutes

LSD Retention Time Comparison

Initial Oven: 100 Celsius, Hold 1 minute, Ramp: 10 Celsius/minute to 325 Celsius, Hold 5 minutes
Run Time: 28.5 minutes

Fluorofentanyl Retention Time Comparison Using Mixed Standard

Oven: 250 Celsius, isothermal (no ramp)
Run Time: 15 minutes

Appendix 3 – Guidance for Specific Identifications

Fentanyl via Mass Spectrometry

To use mass spectrometry as a positive confirmatory test for fentanyl, the following mass spectral features must be observed:

- m/z 334 present
- m/z 348 absent
- m/z 105 roughly twice the abundance of m/z 118
- m/z 119 at least twice the abundance of m/z 119

Selection of "Fentanyl" in the mass spectral comparison findings in the notes means that these features were observed.

Appendix 4 - Notification Regarding Calibration of Reference Masses

Customer Name: State of Alaska – Scientific Crime Detection Laboratory
Shipping Address: 4805 Dr. Martin Luther King Jr. Ave, Anchorage, AK 99507
Customer Number: 907-269-5740 (Front Desk)
Customer Email: Dps.scdl.chemistry@alaska.gov

Hello,

Thank you for taking the time to read this before beginning service!

Enclosed are reference masses for calibration. Our lab recently developed new requirements for external providers including calibration. We have included these requirements on this document to ensure we are clear about service expectations.

Please contact us before beginning service if you have any questions.

Reference Mass Calibration Technical Requirements

- 2 milligram to 500 gram kit masses
  - Troemner UltraClass tolerances
- 1 kilogram, 2 kilogram, and 5 kilogram individual masses
  - ASTM Class 1 tolerances

Calibration Certificate Requirements

Each supplied calibration must be accompanied by a hard copy or electronic calibration certificate. Reference mass calibration certificates must include the following elements:

- Indication that the calibration was performed to ISO/IEC 17025 requirements
- Calibration supplier accrediting body and certificate number
- Test item description or model number
- Nominal mass or mass range
- Calibrated device serial number
- Date calibration was performed
- Date next calibration is due (annual, due at the end of the month)
- Units of grams and kilograms are used
- Condition as of the item as received/assessed
- As Found and As Left results clearly present
- Tolerance/passing limit applied
- Clear indication when As Found testing does not meet specified requirements

- Where adjustment/cleaning was performed, a clear indication that this was done
- Uncertainty associated with each calibration level/mass
  - Must include coverage probability as a percent and k-value(s)
- Calibration traceability information
- Uncertainty statement including k-value(s)

Calibration Sticker Requirements

Calibration stickers reference masses must be applied by the calibration supplier. Calibration stickers for references masses are not applied directly to the masses themselves but rather to their containers. Calibration stickers for this type of equipment must include the following elements:

- Calibration supplier name
- Calibrated equipment serial number
- Date calibration was performed
- Calibration due date (annual, due at the end of the month)

Revision History

Location	Revision made
Throughout	Check for grammar, spelling, word choice, and functionality of links. Removed all references to JetClean module, hydrogen generators, and hydrogen generator maintenance since these items are not being used for Seized Drugs instruments. Broadened Technical Lead-specific approvals to include the discipline supervisor.
Table of Contents	Reduced visible levels to Headings 1 and 2 to shorten
Minimum Criteria	Reworded the requirements around the identification of Cannabis and cannabinoids. Added clarification on samples missing microscopic details sufficient to identify as Cannabis.
Scope of Testing	Added clarification that qualitative analysis will be performed on suspected cannabinoid items if sufficient material is present and item selection would result in testing for that item
Additional Considerations	Added subsections headers and rearranged
Quantitative Analysis for THC	Complete rewrite to reflect outsourcing of this service
Reference Mass Calibration Technical Requirements	Updated to add reference mass calibration class references which are linked to manufacturer accuracy and precision requirements
Storage Conditions and Expiration Dates	Increased some chemical expiration timeframes. This is intended to extend expiration for reagents already in use (where applicable) up to, but not exceeding, the maximum reagent expiration timeframes listed in this manual.
Standard and Reference Material Control	Added hyperlink to the new Seized Drugs Control Number Lists document set
Training Material Control	Added hyperlink to the new Seized Drugs Control Number Lists document set
Drug Standard or Reference Material Breakdown/Degradation	Removed most language – tabled specific clauses for a possible future revision
Labeling Requirements	Clarified that expiration dates need to be applied to chemicals at the time of receipt

Location	Revision made
Qualitative Reporting of Suspected Marijuana or THC Products	Complete rewrite to reflect outsourcing of this service.
Evidence Handling and Marking	Updated section to reflect new Labwide policy requirements.
LSD Retention Time Comparison	Updated method with more appropriate parameters to achieve desired resolution
Appendix 3 – Guidance for Specific Identifications	New Section
Appendix 4 - Notification Regarding Calibration of Reference Masses	New section