

# Alaska Scientific Crime Detection Laboratory

## Controlled Substances Analysis Manual

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### Section 1 Sample Selection

#### Item Selection Policy

In order to provide relevant and timely service the Scientific Crime Detection Laboratory (SCDL) has adopted policies in the Controlled Substances Discipline involving the prioritization and selection of evidence analyzed. If, during pretrial processes, it is determined items that were not analyzed are necessary for prosecution then, upon resubmission, those items will receive top priority by the laboratory. Selection of items for analysis is based on the following criteria:

- Items suspected to contain higher scheduled drugs will receive higher priority.
- If weighable quantities of a drug are present, residues will not be analyzed. Exceptions can occur when the residue present is suspected to be a higher scheduled substance than the weighable quantities.
- If multiple items are submitted that are suspected to be the same substance, only one item will be analyzed. For example, if three items of white, crystalline substance are submitted, only one would be selected for analysis. Exceptions occur when weight thresholds exist in the Alaska Statutes and distribution cases. Items will be analyzed to meet weight thresholds (see Section 2 Quantity Determination) and distribution cases will be decided on a case by case basis in conjunction with the submitting agency and assigned prosecutor's office.
- Probable cause items will be analyzed if notated on the Request for Laboratory Services (RLS) form or otherwise communicated by the submitting agency.
- The above item selection policy will be adopted for each suspect on the RLS form if items can be associated to specific suspects. This information can come from latent print testing of drug evidence or information from the submitting agency. Information from the submitting agency must be documented on the RLS form or otherwise communicated to the lab or the additional items will not be worked.
- Additional circumstances can affect item selection at the discretion of the Chemistry Discipline Supervisor.

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 case notes.

#### Sample Conservation

An unused portion of the original sample will exist in order to allow for subsequent retesting. In cases where only residue amounts are submitted, the residue must be a visible amount and with a quantity sufficient for analysis and reanalysis. While an attempt is made to conserve sample for reanalysis, additional testing performed in the laboratory (such as latent print testing) might destroy any remaining sample.

If the requesting agency and prosecutor determine that testing that would require consumption of the entire sample should be performed a written acknowledgement of consumption from the prosecutor must be documented in the LIMS.

### Determining Populations

Controlled substance analysts rely on their training and experience along with information provided by the submitting agency in determining which items consist of multiple populations and require sub-itemization prior to analysis.

While the analyst is making determinations about population compositions there is no assumption that can be made about the homogeneity of the population unless full testing is performed on each unit within that population or the sampling plan is followed (see Appendix II Sampling Plan).

- Samples submitted by the submitting agency 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.

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### Section 2 Quantity Determination

All balances utilized in controlled substances casework will be calibrated by an external calibration service that is accredited to ISO/IEC 17025:2005 with a scope of accreditation covering applicable calibration requirements prior to use and on an annual basis. Calibration records are stored in the laboratory's Quality Assurance Records. The calibrated weights used in performance checks are calibrated by an external calibration service that is accredited to ISO/IEC 17025:2005 with a scope of accreditation covering applicable calibration requirements on an annual basis. The weight calibration records are stored in the laboratory's Quality Assurance Records.

All balances are checked on a monthly basis with calibrated weights and a check standard. Any member of the Forensic Chemistry discipline may perform the monthly balance performance checks. This is documented on the Balance Performance Check Form which is stored in the controlled substances laboratory space. The information from the performance checks is also documented in the spreadsheet utilized for the ongoing estimate of the measurement of uncertainty. The Mettler Toledo XS2002S model balances have an acceptable tolerance of +/- 0.02 gram. The Mettler Toledo MS16001L model balances have an acceptable tolerance of +/- 0.2 gram. The Mettler AE163 model balance has an acceptable tolerance of +/- 0.01 gram. If a balance does not pass a performance check it will be taken out of service until repaired and calibrated. The check standard is defined in Appendix III for each balance model.

Each week a balance is utilized in casework analysts will perform a performance check with calibrated weights and a check standard. This is documented on the Balance Performance Check Form which is stored in the controlled substances laboratory space. The information from the performance checks is also documented in the spreadsheet utilized for the ongoing estimate of the measurement of uncertainty. If a balance does not pass a performance check it will be taken out of service until repaired and calibrated.

The Mettler Toledo XS2002S model balances are used for weights up to 2050.00 grams. The Mettler Toledo MS16001L model balances are used for weights of 10.0 grams to 8000.0 grams. The Mettler AE 163 model balance is not currently utilized for casework.

If a balance other than the analyst's assigned balance is used, the balance used will be recorded in the case notes. Actual balance readings are recorded in the case notes and on the report with the exception of less than or equal to 0.09 gram. The method of measurement, either net or gross, shall be indicated in the notes.

A gross weight is obtained and recorded in the notes of all items when only a representative sample will be tested. A net weight will be obtained and recorded in the notes on 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, then net weights are not required, but 'residue' will be recorded.

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

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Tablets or capsules will be weighed. Counting of tablets or capsules is optional except when necessary to implement the Alaska Statutes or in the use of the Sampling Plan. Blotter paper and sublingual films will be weighed and dosage units counted and documented in the notes.

Proper weighing techniques:

- Place material into a tared container and obtain a net weight. This will accommodate most drug samples.
- Weigh material directly.
- 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 sum the individual weights (weight by summation). Analyst must show the individual weights and summation in their notes.

Actual balance readings are reported for weights equal to or greater than 0.10 gram. If the weight is equal to or less than 0.09 gram then "below reporting limits" is reported. "Residue" is reported when evidence cannot be weighed.

### Weight considerations

The following are weights/counts listed in the Alaska Statutes. Any analysis must take these weights/counts into consideration and perform analysis accordingly.

Misconduct Involving Controlled Substances						Critical Weights
	6th	5th	4th	3rd	2nd	
Precursors					6 g	6 g
IA			Any		Any (dist)	
IIA			Any	Any (dist)		
IIIA		25 tabs or < 3 g	25 tabs or 3 g	Any (dist)		3 g or 25 tabs
IVA		< 25 tabs or < 3 g	25 tabs or 3 g, Any (dist)			3 g or 25 tabs
VA		< 50 tabs or < 6 g	50 tabs or 6 g, Any (dist)			6 g or 50 tabs
VIA	< 1 oz	1 oz, <1 oz (dist)	4 oz or 25 plants, 1 oz (dist)			1 oz, 4 oz or 25 plants
Spice (IIIA)	< 6 g	6 g	12 g			6 g, 12 g
Bath Salts (IIA)		< 500 mg	500 mg			500 mg

### Section 3 Analytical Scheme

The intent of the controlled substances analysis scheme is to detect and confirm all controlled substances listed in Alaska Statutes 11.71 with the level of specificity required by those statutes. 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."

When performing a controlled substances analysis, the following three goals will be met before issuing a report:

1. Comprehensiveness
  - a. The analysis scheme will be broad enough in scope so that any controlled substances present at significant quantities will be detected before reporting "No Controlled Substances Detected per Alaska Statutes."
  - b. Reference standards and instrumental library spectra will be of sufficient quality and comprehensiveness to meet this goal.
  - c. Extraction techniques and instrumental parameters will be broad enough in scope to preclude false negative results.
  - d. All analysis schemes will include gas chromatography/mass spectrometry.
2. Specificity
  - a. For all reported substances, a secondary test based on a different chemical principle from the first will be used.
  - b. For reported substances known to share similar mass spectral characteristics with other compounds, a secondary 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.
  - c. Drugs known to undergo conversion when heated to GC inlet temperatures (psilocybin, GHB, clorazepate) will be differentiated using a separate, non-converting technique if required for statute interpretation.
  - d. 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.
3. Reproducibility
  - a. A positive result must be reproduced from a separate sampling of the material being tested before reporting.

The following are analytical schemes that meet these three goals. While the analyst has some discretion on what tests to perform and what order to perform them, the specific tests chosen will meet the comprehensiveness, specificity, and reproducibility requirements outlined above.

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### Plant Material/Marijuana Products

1. Obtain weight, volume and/or count as appropriate
2. Perform Morphology and/or Microscopic Analysis
3. Perform Duquenois Levine test (optional)
4. If a positive result is obtained on either microscopic or Duquenois Levine test, confirm the presence of tetrahydrocannabinol with an appropriate extraction followed by GC/MS
5. If microscopic analysis and Duquenois Levine are negative, treat the plant material as a general unknown.

### Manufacturer Marked Pharmaceuticals

1. Obtain a weight, volume and/or count as appropriate
2. Presumptively identify via markings
3. Confirm presumptive identification with an appropriate extraction followed by GC/MS.
4. If unable to identify via markings or presumptive identification is not confirmed, treat as a general unknown.

### General Unknown

1. Obtain a weight, volume and/or count as appropriate
2. Perform preliminary tests as appropriate
3. Perform an appropriate extraction on a separate sample followed by GC/MS. \* Note that for general unknowns one extraction must be done in either a basic extraction or methanol.
4. If preliminary testing and GC/MS have consistent results, the substance is reported assuming the specificity requirements are met.
5. If preliminary test results or GC/MS from initial extraction are inconsistent, a second extraction, different from the first extraction, will be performed and followed by GC or GC/MS as appropriate.

Note: If a non-controlled substance is detected consistently between two different extracts (a preliminary test is not available) there is not a requirement to continue testing in order to report "No Controlled Substances Detected per Alaska Statutes."

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### Section 4 Reagents and Chemicals

Purchased chemicals will be labeled when received with date received and initials of receiver. When opened, chemicals will be labeled with the date opened and initials of opener.

Containers of prepared reagents will be labeled with the chemical's identity, preparer's initials, date of preparation, hazard labels and the expiration date.

#### Prepared Reagents

Preparation: Formulations for preparing routinely used reagents are located in this section. Each reagent made will be documented on the Reagent Preparation Form. Each reagent is named for tracking purposes by name of reagent, date prepared and initials of preparer. For example: QC Mix MMDDYY Initials.

Verification: Verification procedures for routinely prepared reagents are located in this section and documented on the Reagent Preparation Form. Each new batch of reagent that is prepared must be verified prior to use in casework. Verification may be done by the preparer or by another Controlled Substance analyst. The verifier will initial the Reagent Preparation Form for that batch of reagent to certify that the reagent performed as expected.

Reagents are tested at the time of preparation (where applicable) to ensure that they are functioning properly, and the results are recorded in the Reagent Preparation Form.

A Reagent Preparation Form is used to document reagent preparation and verification. It will include:

- Reagent name and amount made
- Date prepared and initials of preparer
- Formulation and lot numbers or laboratory designated unique identifiers where applicable
- Response to verification check
- Storage conditions and expiration date (if any).
- Initials of person verifying reagent (may be same as preparer).

All of the following reagents with the exception of the Marquis reagent, QC Mix, and Internal Standard Injection Solvents will be verified every three months. The results of the verifications will be recorded in the reagent log book.

Commercially prepared ("NIK Kits") test kits may be used in place of laboratory prepared reagents. Manufacturer's supplied instructions will be followed. The lot number of the test kit will be recorded in the case notes. A positive and negative control will be used each day a commercially prepared kit is used and the results of these tests will be documented in the case notes.

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The following are commonly used reagents however other reagents can be used if they have been shown to be acceptable by the scientific forensic community such as those that are listed in the reference book, Clarke, E.G.C. Isolation and Identification of Drugs or other reputable forensic publications and perform as expected with positive and negative controls.

### Quality Control Mixture (QC Mix)

This is the only reagent where the laboratory designated unique identifiers are required to be listed on the Reagent Preparation Form and does not require a hazard label.

**Ingredients:** MSM 10 mg, Cocaine HCl 11 mg, Tetracaine HCl 11 mg, Buprenorphine HCl 11 mg in 10 mL methanol

**Verification Procedure:** Only the four components integrated, resolved, and accurately identified as specified in Section 6 GC and GC/MS Quality Assurance requirements.

**Storage Conditions:** Refrigerated

**Expiration:** 1 year

### Internal Standard Injection Solvent

**Ingredients:** Prepare a 0.5 mg/mL tetradecane solution in the appropriate GC injection solvent.

**Verification Procedure:** GC/MS has only one integrated peak with the mass spectrum of tetradecane.

**Storage Conditions:** Room Temperature

**Expiration:** none

### Borate Buffer

**Ingredients :** 5.4 g NaOH and 20 g Boric acid in 500 mL distilled water

**Verification Procedure:** pH checked with pH paper (between 9-10)

**Storage Conditions:** Room Temperature

**Expiration:** none

### Gold Chloride Crystal Test for Cocaine

**Ingredients:** Solution A: 20% Acetic Acid  
Solution B: 5% H<sub>AuCl</sub>4·3H<sub>2</sub>O in distilled water

**Verification Procedure:** positive (cocaine), negative (blank slide)

**Storage Conditions:** Room Temperature

**Expiration:** none

### Silver Nitrate / Cupric Nitrate Crystal Test for GHB

**Ingredients:** 100 mg of AgNO<sub>3</sub> and 100 mg of Cu(NO<sub>3</sub>)<sub>2</sub> dissolved in 10 mL of water

**Verification Procedure:** positive (GHB), negative (blank slide)

**Storage Conditions:** Room Temperature

**Expiration:** none

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### Marquis Reagent

**Ingredients:** 20 mL concentrated sulfuric acid, 16-20 drops formaldehyde (37%)  
**Verification Procedure:** positive (guaifenesin - purple), negative (blank spot plate)  
**Storage Conditions:** Room Temperature  
**Expiration:** 1 month

### para-Dimethylaminobenzaldehyde (p-DAB or Van Urk's)

**Ingredients:** 1 gram of para-dimethylaminobenzaldehyde in 100 mL ethyl alcohol and 10 mL concentrated HCl.  
**Verification Procedure:** positive (psilocyn/mushroom – violet), negative (blank spot plate)  
**Storage Conditions:** Room Temperature  
**Expiration:** none

### Scott's / Acidified Scott's

**Ingredients:** Solution A: 2% cobaltous thiocyanate in water or 10% acetic acid (acidified Scott's) and diluted 1:1 with 96% USP glycerine  
Solution B: Concentrated HCl  
Solution C: Dichloromethane  
**Verification Procedure:** positive (cocaine salt), negative (blank test tube)  
**Storage Conditions:** Solution A – Refrigerated; Solutions B and C - Room Temperature  
**Expiration:** none

### Weber Reagent

**Ingredients:** Solution A: Add approximately 10 mg of Fast Blue B Salt [o-Dianisidine bis (diazotized) zinc double salt] to approximately 2 mL water. The solution will have a faint straw color.  
Solution B: Concentrated HCl  
**Verification Procedure:** positive (psilocyn mushrooms), negative (blank spot plate)  
**Storage Conditions:** None due to 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 Procedure:** positive (marijuana), negative (blank spot plate)  
**Storage Conditions:** Solution A – Refrigerated; Solutions B and C - Room Temperature  
**Expiration:** none

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### Section 5 Standards

#### Types of Standards

##### Primary Standards

**Definition:** These are compounds whose manner of origin and composition is known and documented. This is typically expressed as: compound name, name of manufacturing organization, lot or batch number and date received. These are compounds purchased from an approved provider (see VENDORS in LIMS for a list of approved providers). Primary standards may be a pure (neat) compound or a solution of a pure compound. All primary standards are verified by instrumental analysis (GC/MS or FTIR) prior to use in casework.

**Maintenance:** Primary standards are retained within the security of the chemistry laboratory. Controlled primary standards are stored in a locked cabinet, refrigerator, or freezer (as appropriate) in the Standards/Chemical Prep Room #2116. Non-controlled primary standards are stored in an unlocked cabinet in the Standards/Chemical Prep Room #2116. Receipt of these standards is logged in the Primary Drug Standards Log Book along with the Certificate of Analysis (if available) and the lab generated confirmation data. See Standard Control for the procedure.

**Use:** Primary standards can be utilized as reference standards in research and development of methods, training, quality control of critical reagents, and for qualitative analysis of casework. Primary standards can 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 can be used to prepare working standards.

##### Secondary Standards

**Definition:** These are compounds whose manner of origin and composition is not documented but exist in the chemistry section as a result of removal from analyzed cases by present or previously employed analysts, from pharmacies or other source of similar nature. All secondary standards should be verified by GC/MS prior to use in casework.

**Maintenance:** Secondary standards are retained within the security of the chemistry laboratory. Controlled secondary standards are stored in a locked cabinet, refrigerator, or freezer (as appropriate) in the Standards/Chemical Prep Room #2116 or in a locker in the Chemistry Evidence Room # 2115. Non-controlled secondary standards are stored in an unlocked cabinet in the Standards/Chemical Prep Room #2116. These standards are logged in the Secondary Drug Standards Log Book along with the lab generated confirmation data. See Standard Control for the procedure.

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

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### Working Standards

**Definition:** These are dilute solutions of primary standards or secondary standards prepared for analytical use.

**Maintenance:** Working standards are retained within the security of the chemistry laboratory. The stock solution of each working standard is stored in the refrigerator in the Chemistry Instrument Room #2117. Subsequent vials may be filled from the stock solution and can be stored in the chemistry laboratory. See Standard Control for the procedure.

**Use:** See primary and secondary standard uses.

### Training Materials

**Definition:** These are compounds that are kept in locked storage in Standards/Chemical Prep Room #2116 and the Chemistry Evidence Room #2115 but are not used for any analytical purpose. Training materials may be taken from case work submissions. When the compound is taken from an analyzed case, the Controlled Substances Discipline Supervisor will be notified and the removal of material will be documented in the case file.

**Maintenance:** An initial inventory of training materials is available and new materials will be added to the inventory as they are introduced to the system but the inventory will not be routinely audited.

**Use:** Training Materials are used for training and displays only.

### Standard Control

#### Traceability

Standards will be traced using a laboratory designated unique identifier. This identifier will include a Control Number and a two letter designation. Examples: 1-AE, 45-AA, 102-EP.

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

Compounds considered standards will be assigned a Control Number. The list of Control Numbers is maintained by the Standards Maintenance Officer and kept with the Primary and Secondary Drug Standards Log Books.

When new compounds are obtained by the laboratory, the list of Control Numbers and the inventory will be updated by the Standards Maintenance Officer.

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

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Each new vial of a compound will be assigned a two letter designation that will follow, in alphabetical order, that of the previous, most recent, vial on hand.

Example: A standard vial of heroin designated AA is consumed. A new vial of heroin is received and designated AB.

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

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

### Primary Standards

#### Receipt of a new primary standard

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

Complete the Drug Standard Control Form.

Place the original standard vial in the appropriate box, alphabetically according to the common compound name, in the locked cabinet, refrigerator, or freezer (as appropriate). Example: diacetylmorphine common name is heroin; the drug standard will be located in the "H" box.

Add the completed Drug Standard Control Form to the Primary Drug Standard Log Book in alphabetical order according to the common compound name. Example: diacetylmorphine common name is heroin so the Drug Standard Control Form will say Heroin (diacetylmorphine HCl) and be located in the "H" section.

#### 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 or FTIR and 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. Only one vial per lot number need be confirmed. Only one vial per lot number will be open at one time.

Standards received in solution will be upheld to manufacturer's expiration date and disposed of in the proper manner.

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### Portion Control

Primary Standards will be kept under the original manufacturer's seal until opened to portion a sample for testing purposes.

All controlled 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.

Gross weights will be recorded. The gross weight will include the container with labels, lid, and contents. The initial weight taken when a vial is first put into service will be done after the manufacturer seal is removed, to provide uniformity for subsequent sampling weights.

### Depletion of Standard Vials

When an original standard vial has been depleted, the corresponding Drug Standard Control Form will be retrieved, initialed, and dated to record when the vial was depleted. The vial itself can be discarded after the form reflects that the standard has been depleted.

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

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

### Secondary Standards

#### Acquisition of a new secondary standard

Initial and date the original standard vial upon receipt. If the compound is taken from an analyzed case, the Controlled Substances Discipline Supervisor will be notified and the removal of material will be documented in the case file. If the compound is taken from an analyzed case or does not come in a vial, prepare an original standard vial and label with compound name, laboratory-designated unique identifier, initials, and date.

Complete the Drug Standard Control Form.

Place the original standard vial in the appropriate box, alphabetically according to the common compound name, in the locked cabinet, refrigerator, or freezer (as appropriate). Example: diacetylmorphine common name is heroin; the drug standard will be located in the "H" box.

Add the completed Drug Standard Control Form to the Secondary Drug Standard Log Book in alphabetical order according to the common compound name. Example:

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diacetylmorphine common name is heroin so the Drug Standard Control Form will say Heroin (diacetylmorphine HCl) and be located in the "H" section.

### Quality Assurance

The standard must be confirmed by GC/MS or FTIR and 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 acquisition of the compound; however it must be performed prior to the first use as a standard.

### Portion Control

All controlled 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.

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

### Depletion of Standard Vials

When an original standard vial has been depleted, the corresponding Drug Standard Control Form will be retrieved, initialed, and dated to record when the vial was depleted. The vial itself can be discarded after the form reflects that the standard has been depleted.

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

### 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 vial, and the date prepared (MMDDYY).

Prepare an approximate concentration of 1 mg/mL in an appropriate solvent of the drug standard. Since these standards are not used for quantitative analysis, approximate concentration is sufficient.

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### Quality Assurance

Working standards must be able to be traced back to the original standard vial they were prepared from at all times and will expire one year from the date prepared.

### 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.

## Log Books and Contents

The Drug Standard Log Books are to be stored in Standards/Chemical Prep Room #2116. Each respective log book will contain the following:

A current inventory of Primary and Secondary standards to be audited annually, no later than September 1.

A Drug Standard Control Form for each Primary and Secondary Standard in the inventory.

The quality assurance data for each Primary and Secondary Standard in the inventory attached to its respective Drug Standard Control Form and a Certificate of Analysis, if available.

The Archived Drug Standard Log Book is to be stored in Standards/Chemical Prep Room #2116. It will contain the following:

A Drug Standard Control Form, with its 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

A folder containing data for archived standards can be located in the LIMS case DRUGS under Archived Drug Standards.

Items in the Archived Drug Standard Log Book will be transferred on a routine basis to the electronic archive.

A cover page will accompany each transfer listing the compounds that can be found in the entry.

Data collected prior to the effective date of CSAM 2013 R0 being implemented can be found in this folder as well.

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### Security

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 Controlled Substances Discipline Supervisor. The Controlled Substances 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 is responsible for assuring that drug standards maintained on the premises are secure.

Access to primary and secondary standards is restricted to the Forensic Chemistry Discipline members, the Quality Assurance Manager, and the Forensic Laboratory Manager. An official memo is submitted, at least annually but updated as appropriate, to the Quality Assurance Manager and the Forensic Laboratory Manager listing by name the individuals with access to the inventory. This document can be located in [I:/Discipline Shares/Controlled Substances/Drug Inventories](#) by year.

If storage of any or all of the primary and/or secondary standards, either temporarily or permanently, is to deviate from the previous mentioned locations, it must be approved by the Controlled Substances Discipline Supervisor.

There are two sets of keys to the locked standards locations and are available for use by the approved personnel. One set of keys is held by the Controlled Substances Discipline Supervisor and the second set of keys is held by the Forensic Laboratory Manager.

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

An audit of all primary and secondary standards will be made annually, no later than September 1, updates performed, and an official memo written to the Quality Assurance Manager and the Forensic Laboratory Manager indicating that this audit was completed. The audit will account for all primary and secondary standards within the laboratory and will include a yearly gross weight for each controlled substance. This document can be located in the LIMS case DRUGS under Annual Audits.

### Section 6 Analysis Techniques

#### COLOR TESTS

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

##### MARQUIS TEST

Place 1-3 drops of Marquis Reagent in a clean white spot plate or test tube along with the sample; observe and record response.

Common Responses:

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

##### SCOTT'S TEST (Cobalt Thiocyanate)

Place sample in a small tube. Add approximately 5 drops of Scott's Solution A to sample and shake. Observe the formation of blue color and/or precipitate. Cocaine salts give a clumpy blue precipitate while cocaine base gives no reaction at this step.

Add approximately one drop of Scott's Solution B. Solution will turn pink and any blue color from step one will disappear.

Add several drops of Scott's Solution C and shake. The lower layer will develop an intense blue color if cocaine (salt or base) is present.

Cocaine gives a positive test, which is a pink layer over a blue layer on the final step.

##### ACIDIFIED SCOTT'S TEST (Cobalt Thiocyanate)

This modification of the Scott's Test uses 10% acetic acid instead of water in the preparation of the Solution A. Since the acid converts cocaine base to its salt form. Both forms will give a positive reaction (turn blue) to the first step of this test.

### 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. Add Weber Solution A and look for a purplish-red color. Then add one drop of Weber Solution B. A blue color indicates psilocyn, while no color occurs with psilocybin.

### DUQUENOIS-LEVINE

Add 1 volume of Duquenois-Levine Solution A to the sample and shake. Then add 1 volume of Duquenois-Levine 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 Duquenois-Levine Solution C and note whether the color is extracted into the bottom layer. If positive for THC, a blue-violet color will be extracted into the lower layer.

### CRYSTAL TESTS

Crystal tests are preliminary in nature. The following crystal tests are commonly used:

#### GOLD CHLORIDE (Cocaine)

**Procedure:** Place sample on a glass slide and add 1-2 drops Gold Chloride Solution A. Add one small drop of Gold Chloride Solution B and observe on the polarizing microscope. A result of positive for cocaine in the notes indicates the appearance of long rods with one or many arms at nearly right angles to the main axis.

#### SILVER NITRATE/CUPRIC NITRATE (GHB)

**Procedure:** Place sample on glass slide and add 1 or 2 drops of reagent. View crystals with a polarizing light microscope. Rectangular crystals grow at the edges in under 5 minutes are positive for GHB.

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### PHYSICAL IDENTIFICATION OF PHARMACEUTICALS

Pharmaceutical preparations can be presumptively identified with 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. Triaging of multiple items in a case has identified the pharmaceuticals as not apparently vital to the total prosecution.

The following are accepted as references for use in establishing physical identification of pharmaceuticals:

- Physician's Desk Reference (PDR)<sup>TM</sup>, Medical Economics
- Logo Index<sup>TM</sup>, DEA
- Identidex<sup>TM</sup>, Micromedex Inc.
- Drug Identification Bible<sup>TM</sup>, Amera-Chem, Inc.
- Government and manufacturer's websites (Printed documentation of the pharmaceutical reference must be included in the case notes when using a website as a reference. For example a screenshot of the webpage with the date accessed in the notes.)
- Ident-A-Drug<sup>TM</sup>, Therapeutic Research Center
- Pharmer.org, Drugs.com, RxList.com, or other information websites
- In-house Pharmaceutical Identification Reference

### VISUAL ANALYSIS OF PLANT MATERIAL

Macroscopic Examination: The entire sample should be visually checked for homogeneity. A positive result indicates that the identifying characteristics of marijuana are present, such as:

- palmate, serrated leaves
- flowering bud material
- seeds ovoid in shape
- stems are squared in cross-section

If the macroscopic examination is negative for marijuana there is no need to perform a microscopic analysis.

#### 1. Plant material

- a. Observe sample under stereomicroscope.

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

#### 2. Resin extract of Marijuana

- a. Observe sample under stereomicroscope and/or compound microscope.  
Microscopic examination may reveal cystolithic hairs or clothing hairs.

Germination of seeds to demonstrate viability is not performed.

### 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 instrumental analysis. Information as to solubility and specific physical properties can usually be found in *Clarke's Isolation and Identification of Drugs and Poisons*. The choice of an organic solvent is dependent upon the drug to be extracted and the preference of the analyst.

#### Residue Collection

- Paraphernalia with visible residue may be:
  - Rinsed with internal standard injection solvent
  - 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 an internal standard injection solvent that dissolves the drug of interest
  - 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 internal standard injection 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 internal standard injection 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 internal standard injection solvent.
    - Vortex or shake.

#### Other extractions available

- Food Products with suspected THC
  - Place a portion of sample in a disposable test tube
  - Add hexanes
  - Vortex or centrifuge
  - Transfer hexanes to a new disposable test tube

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- 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 hexane
- Acidify using 1N HCl to pH 1-2
- Extract with Hexanes with internal standard added
- Vortex or shake
- Resulting Layers:
  - Top Hexanes Layer – THC if present
  - Acidic methanolic layer – Discard
- Gammabutyrolactone (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, take up in water and then extract with dichloromethane with internal standard.
  - Vortex or shake.
  - Let settle
  - Resulting Layers:
    - GBL/BD will be in the dichloromethane layer.
    - If gammahydroxybutyric acid (GHB) is present, it will be in the aqueous layer.
- Gammahydroxybutyric Acid (GHB) Extraction. Perform a microcrystalline test and then if positive proceed with the following protocol:
  - Remove a portion of sample (or take the aqueous layer from “GBL or BD Extraction” and evaporate to dryness. Keep dried sample at ~105° C
  - GHB derivatization procedure using a derivatizing agent
    - Place the derivatizing agent in three auto-sampler vials or auto-sampler vials with glass inserts.
    - Add a couple of mg on unknown sample to the first vial. To the second vial add a couple of mg of the standard GHB. Leave a third vial blank with only derivatizing agent.
    - Derivatize capped vials at 90° C for 10 minutes (hotplate or warm water bath).
- Mushroom Extraction
  - Grind mushrooms 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 and vortex
  - Centrifuge sediment to bottom and transfer acidic ethanol to a test tube
  - Evaporate to dryness
  - Reconstitute with dichloromethane with internal standard, vortex, and analyze by GC/MS
- Clorazepate Extractions
  - Extraction Method 1
    - Place powder from a capsule or crushed tablet in a container
    - Add approximately 3 mL of 15N ammonium hydroxide
    - Stir the mixture

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- Centrifuge or allow to settle
- Remove the aqueous layer and evaporate the resulting residue is the clorazepate salt, suitable for FTIR analysis
- Extraction Method 2
  - Place powder from a capsule or crushed tablet in a container
  - Add approximately 12-15 mL of dichloromethane and methanol mixed in a 3:1 ratio
  - Mix vigorously
  - Centrifuge and allow to settle
  - Remove the liquid and filter it through a filter presoaked with the dichloromethane:methanol mixture
  - Add 1.5 to 2 mL of water to the filtrate and mix thoroughly
  - Centrifuge or allow to settle
  - Remove the aqueous top layer and wash it twice with dichloromethane, centrifuging after each wash. (If the dichloromethane layer contains a white opaque foam-like substance after the second wash, repeat the wash until the dichloromethane layer is clear.)
  - Evaporate the aqueous layer. The resulting residue is the clorazepate salt, suitable for FTIR analysis.

### GAS CHROMATOGRAPHY

#### Materials

Electronic copies of manufacturer's instrument manuals are located on the laboratory's network drive.

The following GC column stationary phases are approved for use in the controlled substances discipline:

- 100% Dimethylpolysiloxane
- (5%-Phenyl)-methylpolysiloxane (or equivalent)
- (50%-Phenyl)-methylpolysiloxane (or equivalent)

The column parameters used are:

- 30 m length
- 0.25 mm inner diameter
- 0.25 um film thickness.

The specifications for the column used on a particular instrument will be recorded in the printed method located in the instrument's maintenance binder.

With the exception of derivatizing agents, GC injection solvents used for case samples and their respective negative controls will contain an internal standard (tetradecane at 0.5 mg/mL). Solvents used to prepare working standards will not contain an internal standard.

Hydrogen generators provide the hydrogen gas for flame ionization detectors.

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### Instrument Parameters

The SCREEN.M method is used when analyzing samples for routine casework. Refer to the GC and GC-MS Working Instructions for a table of the GC parameters used in this method.

Copies of the method parameters used by each gas chromatograph are stored in their respective instrument maintenance binders. No permanent changes will be made to currently used GC methods without approval from the discipline supervisor. When a change is made, a copy of the previous method parameters will be imported into the LIMS and marked with its start and end dates. Temporary changes to split ratios and oven program parameters are allowed when appropriate. These changes will be documented in the case file(s) in which the temporarily modified method was used.

### Analysis

Each analyst is responsible for ensuring that the instrument has passed all quality assurance requirements before analyzing case samples. All samples will be analyzed using the SCREEN.M or SCREEN20.M method. A negative control prepared in the same manner and on the same day as the case sample will be analyzed directly prior to the case sample. In situations where multiple case samples are prepared in the same manner and analyzed on the same day, the same prepared negative control can be used in each analysis. For retention time comparison, a standard must be analyzed using the same method conditions as the sample within 24 hours of when the sample was analyzed.

The instrument software is programmed to analyze a series of samples through a sequence table. The GC and GC-MS Working Instructions have procedures describing the details of this process.

Results from each sample acquisition are electronically printed to the "JT Print" folder located on the desktop of the instrument computer. These images are transferred to their appropriate case files in the LIMS.

### Acceptability Criteria

An acceptable blank is one that results in no integrated peaks other than the internal standard. When performing a retention time comparison, the integrated retention times of the case sample analyte and the comparison standard must be within 0.05 minutes for the test to be considered positive.

### Quality Assurance

On the first working day of the week, the QC Mixture must be analyzed on the gas chromatograph before it can be used for case work. Also, whenever maintenance has been performed on the gas chromatograph, the QC Mixture must be analyzed to ensure the instrument is working properly. Passing criteria for the QC Mixture are:

- All components of the mixture are adequately separated and integrated
- No excessive fronting or tailing of peaks is observed
- No extraneous peaks are integrated

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- The abundance and retention time of each peak is consistent with previous analyses of the same QC Mixture lot on that instrument. Any shift in retention time due to column trimming or changing will be documented in the maintenance log section of the instrument maintenance binder.

The person running the QC Mixture will place their initials in the comment field. The person approving the passing criteria have been met will place their initials and date on the first page of the printed QC Mixture stored in the maintenance binder.

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. Maintenance is addressed in the Controlled Substances Working Instructions manual.

Hardcopies of passing QC Mixture results will be kept in the corresponding instrument maintenance binder. At least twice a year, the previous year's QC data will be scanned and entered into the LIMS case file CS INST YYYY under the appropriate instrument and the hardcopies will be destroyed.

### MASS SPECTROMETRY

The section on gas chromatography covers the GC component of GC-MS whereas this section specifically covers the MS component.

#### Materials

Electronic copies of manufacturer produced instrument manuals are located on the laboratory's network drive.

#### Instrument Parameters

To ensure consistent total ion abundance and relative ion abundance between instruments, the standard tune (stune.u) program will be used when tuning a mass spectrometer used for controlled substances casework.

Copies of the MS method parameters used by each GC-MS are stored in their respective instrument maintenance binders. With the exception of changing the solvent delay due to column maintenance, no permanent changes will be made to currently used MS methods without approval from the discipline supervisor. When a change is approved, a copy of the previous method parameters will be imported into the LIMS and marked with its start and end dates.

Temporary changes to MS scan ranges are allowed when appropriate. These changes will be documented in the case file(s) in which the temporarily modified method was used.

The GC-MS software is programmed to automatically integrate detected peaks and perform library searches of their mass spectra. The integration parameters used are listed in the Controlled Substances Working Instructions manual.

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Spectra obtained from analyzing working standards may also be used when making comparisons. When a working standard that has not been previously added to the in-house library is used, its mass spectrum will be added as described in the Mass Spectral Libraries section.

### Mass Spectral Libraries

The discipline's in-house library (SCDL.L) and/or outside libraries from reputable sources may be used during mass spectral searches. The library search report will list which libraries were used for each sample analyzed. Libraries will be located on each instrument computer 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 SCREEN.M method. 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 also be included.

Whenever a new entry is added to the discipline's in-house library, the updated version will be copied to each instrument computer and replace the previous version. The contents of outside libraries will not be edited in any way, however if a newer version of an outside library is obtained, it will replace the previous version on every instrument computer.

### Working Instructions

All procedures referred to in the GC section will be followed when performing a GC-MS analysis. Each analyst is responsible for ensuring that both the GC and the MS have passed all their quality assurance requirements before analyzing case samples.

### Acceptability Criteria

It is permissible to use GC/MS integrated retention times for GC retention time data. Acceptability criteria for this test are outlined in the Gas Chromatography section.

The mass spectrum of each sample is visually compared with that of a known standard or reputable library. The significance of peaks (both absent and present) is noted and no prominent ions should be missing from the evidence spectrum. For a match to be considered acceptable the major ions should agree between unknown and standard and the presence or absence of a 'molecular ion' must agree between unknown and standard. Due caution will be made when a library search result gives matches of different compounds with very similar mass spectra (see Analysis Scheme section).

If GC/MS data is rejected, the reason for the rejection will be recorded in the notes and the spectra saved, in addition to the non-rejected data.

### Quality Assurance

On the first working day of the week, mass spectrometers must be tuned before they can be used in case work. The mass spectrometer must also be tuned with the stune.u program

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whenever it has been cleaned or serviced prior to resuming case work. Passing criteria for a standard tune are:

- The ion abundances of 18 (water), 28 (nitrogen), and 32 (oxygen) relative to ion 69 are less than 10%
- The three principle peaks (69, 219, and 502) have smooth and symmetrical 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. Any maintenance that occurs will be recorded in the instrument's maintenance binder. The mass spectra of the QC Mix components will also be reviewed. To be considered acceptable, accurate library matches must be made for each component of the QC Mixture.

Hardcopies of passing tune results will be kept in the corresponding instrument maintenance binder. They will be filed chronologically with the QC Mixture results and archived using the same procedure outlined in the GC section. Automated library search results for the QC Mixture will be included with the chromatogram hardcopy.

### INFRARED SPECTROPHOTOMETRY (FTIR)

A FTIR Spectrometer with an ATR (attenuated total reflectance) accessory for analysis of drug substances is used.

Electronic copies of manufacturer produced instrument manuals are located on the laboratory's network drive.

All samples will be analyzed using the ATR.exp.

Procedures for FTIR analysis:

- Collect a background spectrum (blank) of air.
- Acquire a blank spectrum of the ATR crystal.
- Place small amount of sample on ATR crystal.
- Acquire sample absorbance spectrum and search libraries to assist with identification.

If significant amounts of interfering substances are present, extract the sample using any extraction which successfully isolates the substance of interest. Use caution to prevent conversion between base and salt forms when this is an issue.

The case file will include the blank and sample spectra along with any library or standard spectra used for identification. If FTIR sample data is rejected, the reason for the rejection will be recorded in the notes and the spectra saved, in addition to the non-rejected data.

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### Interpretation of Data and Criteria for Identification by FTIR analysis:

- For identifying a reportable drug, the sample spectrum must be visually compared with the spectrum of a standard, either run on the same instrument using the same sampling mode (ATR), or the sample spectrum must be visually compared to a library-generated spectrum.
- Unknown materials may contain extra absorbance bands due to sample impurities.

The significance of absorbance band peaks (both absence and presence) and relative intensities of absorbance bands should be assessed. However, no prominent bands should be missing from the unknown spectrum.

In-house and/or outside libraries from reputable sources may be used during spectra searches. Examples are:

- HR Georgia State Forensic Drugs
- Alaska Scientific Crime Detection Laboratory library

Copies of the ATR.exp are stored in the instrument binder. With the exception of temporarily changing the number of scans, no changes will be made to the currently used method without approval from the discipline supervisor. When a change is approved, a copy of the previous method parameters will be imported into the LIMS and marked with its start and end dates.

### Quality Assurance

In the first week of the month the monthly quality assurance will be performed.

- Acquire a background spectrum (blank) of air.
- Acquire spectrum from a polystyrene standard. Check for absorption at  $1601\text{ cm}^{-1}$ ,  $\pm 2\text{ cm}^{-1}$ . This peak will be labeled. The polystyrene spectra should give a correlation match of 90 or greater with library spectra. Record the polystyrene results in the FTIR computer notebook and place spectra into the instrument binder.
- Instrument validations are run after any major service or hardware replacement. The manufacturer's software program will be used to determine performance of wavenumber accuracy, resolution, and signal-to-noise. The field on these reports for annotating the scientist performing the test, date, and verification by another scientist need not be filled out since this information is satisfied by the name and date appearing on the document.
- Record system suitability and performance verification reports, and polystyrene standard spectra is accomplished on or about the first working day of the month. Hard copies are compiled in the FTIR log book and then annually placed in LIMS in the folder CSINST XXXX.

Records: The following documents must be maintained in the laboratory for the Infrared Spectrometer. Some data may be duplicated between hard copy in the log book and electronic storage in LIMS:

- Description of the instrument system
- Documentation of polystyrene performance data
- Documentation of validation checks
- Documentation of all maintenance and repairs

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### Section 7 Reporting

All controlled substances identified will be reported by the language listed in the Alaska Statutes.

When a controlled substance is not detected the report will state: "No Controlled Substances per Alaska Statutes detected."

Analyst may report a non-controlled substance identified. This will be listed after "No Controlled Substances per Alaska Statutes detected." And state: "\_\_\_\_\_, a non-controlled substance was identified."

When a small sample is present and there is not enough sample for complete testing while maintaining a portion untested the report will state: "Quantity insufficient for analysis."

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

When only a physical identification of a pharmaceutical product by literature reference is performed the report will state: "Not Analyzed. Markings indicate \_\_\_\_\_ a controlled (non-controlled) substance per Alaska Statutes."

When a non-controlled substance in a controlled pharmaceutical product does not affect the scheduling of the controlled substance, the report will only list the controlled substance.

When a pharmaceutical product contains only non-controlled substances, the report will state the non-controlled substances.

When the non-controlled substance in a controlled pharmaceutical product does affect the scheduling of a controlled substance, the report will state the non-controlled substance: "Not Analyzed. Markings indicate \_\_\_\_\_ and \_\_\_\_\_ a controlled mixture per Alaska Statutes."

Any alternative wording on reports is not permitted without prior approval from the discipline supervisor.

In order to clearly state on reports what analysis was performed, items tested will be sub-itemized on the report from items of the same population that were not tested. Below are two examples:

Description

Item 1 Blue tablets

Item 1a One blue tablet

Result

Item 1 Not Analyzed. Markings indicate hydrocodone, a controlled substance per Alaska Statutes.

Item 1a Hydrocodone, net weight 0.25 gram +/- 0.03 gram.

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### Description

Item 1 Black plastic bags

Item 1a One black plastic bag containing white powder

### Results

Item 1 Not Analyzed.

Item 1a Cocaine, net weight 0.28 gram +/- 0.03 gram.

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### Section 8 Administrative

#### Evidence

All evidence transfers are documented in the LIMS. Upon receipt of evidence utilizing a mobile storage device the analyst will transfer the evidence into their personal custody. The analyst will ensure all evidence has transferred properly in the LIMS and ensure the mobile storage device has been cleared electronically in the LIMS.

Personal evidence lockers are provided for each analyst in the controlled substance laboratory and additional larger storage areas are available in the controlled substance evidence room # 2115. Each analyst will ensure all evidence is properly stored prior to leaving the laboratory.

The reagent prep room #2116 has access limited to the Forensic Chemistry personnel.

#### Bench Notes

The bench notes will include at a minimum:

In the analyst's opinion, any significant discrepancies between the request for laboratory services form and the evidence received. Discrepancies on the number of items received must also include a witness by another laboratory member. The person witnessing the discrepancy will log into the LIMS and make a note in the case activities area of the case. The analyst will also email the supervisor the case number to review the discrepancy found.

The start and end dates of analysis. This is recorded by editing the request and entering the two dates in the ASSIGNOR BLOCK.

A detailed description of the item's packaging and physical description of the items received. This must be in enough detail to properly convey the information to the technical and administrative reviewer.

The weight of each item analyzed. See Section 2 Quantity Determination for details.

The sampling plan applied when appropriate.

The sample preparations or extractions used.

Descriptions of each analysis performed with results.

The conclusions reached for each item analyzed.

#### Evidence Marking and Seals

All items analyzed will be marked by the analyst with the case number, the item number and the analyst's initials. This may be accomplished by placing the analyzed item in a marked laboratory bag or marking the bag it was received in. All items analyzed with a pending latent print request will be repackaged and labeled appropriately.

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All packaging layers received in a sealed condition will be resealed by the analyst. The outer packaging must be sealed with evidence tape, initialed and dated by the analyst crossing the barrier of the seal and the package. Heat sealing is not utilized in the controlled substances discipline as the only form of a seal.

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

### Reports

A report will be issued for each case analyzed. Each report will clearly communicate the items received and analyzed. At a minimum the report will include:

- a brief description of the item(s) analyzed
- weight of controlled substances identified
- count of controlled substances where required for Statute interpretation
- sampling plan if used
- result, conclusion or opinion of the item(s) analyzed

### Amended reports

An amended report will be issued when an error is discovered after the report has been distributed. The case analyst will notify the supervisor and issue an amended report following the Quality Assurance Manual 5.10.9 will be issued.

### Reviews

Each case will be technically and administratively reviewed prior to distribution. The minimum requirements listed in the Quality Assurance Manual will be completed. In addition the technical reviewer will ensure:

- The imaging file names match the spectra
- Standards are properly documented

The 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 activities and will include a disclaimer that this is preliminary information. The Quality Assurance Manual outlines further details on the release of preliminary results under 5.10.3.3.

Discovery for the controlled substances discipline is managed by the discipline supervisor other than routine bench note requests.

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### Validation requirements

When new instrumentation is acquired, the manufacturer's representative will install and perform the initial set up of the instrument. The documentation will be stored with the discipline records in the LIMS.

Prior to use in case work a validation of the performance of the instrument will be performed. This documentation will be stored with the discipline records.

The minimum requirements for the performance validation are analysis of ten known reference standards appropriate to the new instrument. The discipline supervisor will approve the standards selected for the performance validation. Each reference standard will be run utilizing the approved current casework method. The performance validation data will be stored in the discipline records for the life of the instrument. The performance data will also be forwarded to the Quality Assurance Manager.

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**Appendix I Abbreviations**

**APAP:** Acetaminophen (for *N*-acetyl-**para**-aminophenol)

**ATR:** Attenuated Total Reflectance

**BB DCM:** Borate Buffer / Dichloromethane

**DCM:** Dichloromethane (Methylene Chloride)

**DIB:** Drug Identification Bible

**ICFI:** Insufficient Concentration for Identification

**ISFI:** Insufficient Sample for Identification

**ID:** Identification

**MSM:** Methylsulfonylmethane (Dimethyl Sulfone)

**N/A:** Not Applicable

**NA:** Not Analyzed

**NCSD:** No Controlled Substances Detected

**PDR:** Physicians' Desk Reference

**QIFA:** Quantity Insufficient for Analysis

**QNS:** Quantity Not Sufficient

**REF:** Refrigerated

**RT:** Room Temperature

**TABS:** Tablets

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### Appendix II Sampling Plan

#### Hypergeometric Sampling Plan\*

Based on statistical probability, there is 95% confidence that at least 90% of the units contain the drug.

The use of the sampling plan requires that all units appear to be homogenous.

If any results are different than the rest, the analyst must re-evaluate the population.

The report will reference the sampling plan utilized and the confidence level and corresponding inferences of the population. The report will state what was received and clearly state the results/conclusions. The weight of the units tested will be reported.

Description

Item 1 163 bags containing plant material \_\_\_\_grams, gross weight

Result

Item 1 A hypergeometric sampling plan was used. Item 1 contains \_\_\_\_ with a 95% confidence that at least 90% of the population contains \_\_\_\_." Weight of tested items

Population Size	Sample Size
1-12	All
13-20	12
21-30	15
31-40	18
41-50	19
51-60	20
61-70	21
71-80	22
81-100	23
101-130	24
131-160	25
181-210	26
211-270	27
471-1000	28

*\*Information is taken from enfsi-dwg sampling calculator 2012 referenced in the ASCLD/LAB Policy on Sampling, Sampling Plans and Sample Selection.*

### Appendix III Uncertainty of Measurement

The estimation of the uncertainty of measurement of the controlled substances weights has been performed. The initial study performed in 2013 has been documented in two reports titled "Controlled Substances Estimate of the Uncertainty of Measurement December 18, 2013 Mettler XS2002S balances and Mettler Toledo MS16001L balances". These reports are stored in the internal network drive of the controlled substances discipline share folder and a copy is kept in the Quality Assurance Records.

The report addresses the following elements:

- Statement of the measurand
- Statement of the measurement traceability
- Equipment used
- 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, the coverage factor, the coverage probability, and the resulting expanded uncertainty
- The schedule to review and/or recalculate the measurement of uncertainty.

Based on this initial study the expanded combined standard uncertainty at 95% coverage for the Mettler Toledo XS2002S balances is +/- 0.03 gram and +/- 0.3 gram for the Mettler Toledo MS16001L balances rounded to one significant figure when used at the Alaska Scientific Crime Detection Laboratory.

These reports also address uncertainty of combined weights utilizing a root sum of squares formula. The expanded combined standard uncertainty at 95% coverage is equal to +/- 0.03 gram x the square root of the number measurement made ( $\sqrt{N}$ ) for the Mettler Toledo XS2002S balances and +/- 0.3 gram x the square root of the number measurement made ( $\sqrt{N}$ ) for the Mettler Toledo MS16001L balances. This will be applied when weight by difference or weight by summation is performed. If the value obtained from the  $\sqrt{N}$  is greater than the readability of the balance utilized, the value will be rounded to the readability of the balance. The value will be rounded up at a 5 or higher and rounded down at a 4 or lower.

An ongoing estimation of the uncertainty of measurement is performed on a weekly and monthly basis. Monthly each balance used in casework will be performance checked with calibrated weights listed on the Balance Performance Check Form and a check standard. Each week a balance is utilized in casework analysts will perform a performance check with calibrated weights listed on the Balance Performance Check Form and a check standard. This information is documented on the Balance Performance Check Form which is stored in the controlled substances laboratory space. The information from the performance checks and check standards is also documented in the spreadsheet utilized for the ongoing estimate of the measurement of uncertainty stored in the internal network drive of the controlled substances discipline share folder.

# Alaska Scientific Crime Detection Laboratory

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## Controlled Substances Analysis Manual

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The check standard chosen for the Mettler Toledo XS2002S balances is tablets packaged for stability. The check standard chosen for the Mettler Toledo MS16001L balances is a powder packaged for stability.

The data from this ongoing estimation of the uncertainty of measurement will be reviewed and an updated estimation of the uncertainty of measurement reports will be written at least annually or if any significant change in the expanded uncertainty is detected.

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# Alaska Scientific Crime Detection Laboratory

## Controlled Substances Analysis Manual

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### Appendix IV Revision History

2013 R3	2013 R2	Location	Revision made
all	all	all	spacing, grammar updated as needed
4	4	Section 2 First Paragraph	First and third sentences changed to specify the vendor used for calibration of the balances and weights.
4	4	Section 2 Second Paragraph	Added "and a check standard." to the end of the first sentence.
4	4	Section 2 Second Paragraph	Removed this sentence "The Balance Performance Check Form details the weights utilized for monthly checks and the tolerance allowed" and replaced with "Any member of the Forensic Chemistry discipline may perform the monthly balance performance checks. The information from the performance checks is also documented in the spreadsheet utilized for the ongoing estimate of the measurement of uncertainty. The Mettler Toledo XS2002S model balances have an acceptable tolerance of +/- 0.02 gram. The Mettler Toledo MS16001L model balances have an acceptable tolerance of +/- 0.2 gram. The Mettler AE163 model balance has an acceptable tolerance of +/- 0.01 gram. The check standard is defined in Appendix III for each balance model."
4	4	Section 2	Added "Each week a balance is utilized in casework analysts will perform a performance check with calibrated weights and a check standard. This is documented on the Balance Performance Check Form which is stored in the controlled substances laboratory space. The information from the performance checks is also documented in the spreadsheet utilized for the ongoing estimate of the measurement of uncertainty. If a balance does not pass a performance check it will be taken out of service until repaired and calibrated."
4	4	Section 2	Removed "High capacity balances will not be used for weighing below 10 grams and analysts should be aware of the upper and lower limitations of the balance used." and replaced with "The Mettler Toledo XS2002S model balances are used for weights up to 2050.00 grams. The Mettler Toledo MS16001L model balances are used for weights of 10.0 grams to 8000.0 grams. The Mettler AE 163 model balance is not currently utilized for casework."

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2013 R3	2013 R2	Location	Revision made
4	4	Section 2	Removed "All weights listed on the report are net weights unless otherwise indicated." Added "notes and on the report with the exception of less than or equal to 0.09 gram." to the sentence "Actual balance readings are recorded in the case notes."
5	4	Section 2 Last Paragraph	Removed "For reporting purposes, weights will routinely be truncated to one decimal point (tenths of a gram). Weight measurements under 0.10 gram will be reported as less than a tenth of a gram or as "< 0.1 g". Added "Actual balance readings are reported for weights equal to or greater than 0.10 gram. If the weight is equal to or less than 0.09 gram then "below reporting limits" is reported. "Residue" is reported when evidence cannot be weighed."
24	24	Section 6 Analysis Techniques	Under the Analysis portion, added "or SCREEN20.M" to the second sentence ". All samples will be analyzed using the SCREEN.M method."
29	29	Section 7 Reporting	Added "When a non-controlled substance in a controlled pharmaceutical product does not affect the scheduling of the controlled substance, the report will only list the controlled substance. When a pharmaceutical product contains only non-controlled substances, the report will state the non-controlled substances. When the non-controlled substance in a controlled pharmaceutical product does affect the scheduling of a controlled substance, the report will state the non-controlled substance: "Not Analyzed. Markings indicate _____ and _____ a controlled mixture per Alaska Statutes."
29 & 30	29	Section 7 Reporting	Example report language results were changed to reflect new reporting weight and uncertainty.
36 & 37	35	Appendix III Uncertainty of Measurement	Removed "The controlled substances category will become compliant with the Supplemental Requirements of the American Society of Crime Laboratory Directors/Laboratory Accreditation Board with respect to measurement of uncertainty when required by the accrediting body." All language in Appendix III is new.