

Scientific Crime Detection Laboratory

Drug Chemistry Procedure Manual

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SECTION 1 INTRODUCTION

1.1 OVERVIEW

The Controlled Substances discipline performs analysis of materials suspected of containing controlled substances, including pharmaceuticals and clandestine lab samples. The *objective* of the analyst is to conclusively identify the presence or absence of controlled substances in a sample.

Controlled substances are those substances designated by the legislature of the State of Alaska in the Alaska Statutes 11.71.

Additionally, Forensic Scientists may be called upon to analyze samples for federal agencies operating within Alaska for substances controlled under the Uniform Controlled Substances Act. This laboratory does not perform purity (quantitative) testing, optical isomer determination, nor does it routinely sample multiple items based on federal threshold levels.

1.2 SAMPLE PRIORITIZATION

In order to provide relevant, timely, accurate service, the Scientific Crime Detection Laboratory (SCDL) has adopted policies in the Controlled Substances discipline involving the prioritization and selection of evidence analyzed:

- If weighable quantities of material are present, residues may not be analyzed.
- If multiple quantities of materials are submitted, one exemplar from each type of item may be analyzed (for example: if 3 bags plant material, 3 bags of chunky material, and 3 bags of powder are submitted, then 1 bag each of plant material, chunky material, and powder may be analyzed).
- If there are multiple suspects, the submitter must indicate which items are associated with which suspects to ensure analysis.
- If the customer has a need for an exception to this practice, this information shall be noted on the request for laboratory service (RLS) by the customer, or documented in the case record if by other means (e-mail, telephone call).

1.3 SAMPLE CONSERVATION

An unused portion of the original sample will be left in order to allow for subsequent retesting. In cases where only residue amounts are submitted, that residue must be a visible amount, in a quantity sufficient for analysis and reanalysis. If a sample does not

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meet this requirement, it will be reported as "Quantity Insufficient for Analysis" with details recorded in the analyst's notes.

When sufficient analytical data exists to suggest that a substance is present, but insufficient sample exists to definitively identify the substance, "Insufficient quantity for complete identification" or "Testing indicates the presence of XXXX. Insufficient quantity for complete identification" will be reported.

Sample vials may be retained in cases where other laboratory analyses may prevent future testing of remaining trace evidence. While retaining the sample vial is an attempt to preserve the sample, it is not a guarantee against sample degradation or leakage.

1.4 IDENTIFICATION REQUIREMENTS

A minimum of two tests performed on independent samples, with positive results pertinent to the substance being reported, with at least one test being mass spectroscopy or infrared spectroscopy, shall be required to conclusively identify a substance, whether controlled or uncontrolled. One sampling, usually done as presumptive testing, can be any of the protocols discussed in this manual:

- Literature reference
- Color test
- Microcrystalline test
- GC/MS
- FTIR

A commercial preparation may be identified by reference material only (for example by manufacturer logo), but shall clearly indicate in the report that identification was by "physical identification only, no chemical testing was performed."

Marijuana (Cannabis) is identified through physical plant characteristics (botanical identification) and GC/MS (chemical identification). The GC/MS must demonstrate the presence of tetrahydrocannabinol.

It is permissible to use the same instrumental technique on two separate samplings to identify a sample.

Special Considerations:

- For salt vs. base determinations of cocaine, infrared analysis is utilized
- For samples indicating the presence of Clorazepate or Nordiazepam refer to the Clorazepate extraction procedure. These drugs cannot be identified by GC/MS and must be extracted and analyzed by FTIR.

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- For mushroom cases a Weber test must be performed to distinguish between psilocyn and psilocybin.
- For negative samples, the testing process between samplings shall be different enough to minimize the possibility of a false negative. Example, a sample may be analyzed by GC/MS with a simple dilution in solvent, and a second sample extracted from buffer to solvent, to ensure a salt, acid, or basic form of a substance is not missed.

The major limitation in the analysis of an item is the size and condition of the sample submitted for analysis.

References

1. Butler, W.P. "Methods of Analysis for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs", Internal Revenue Service Publication No. 341: Washington, D.C., 1967, pp.92-93.
2. Moffat, Osselton, & Widdop, "Clarke's Isolation and Determination of Drugs and Poisons", 3rd Edition, Pharmaceutical Press: London, 2004.

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SECTION 2 TECHNICAL PROCEDURES

2.1 VISUAL SCREENING

When numerous individual 'units' are included in an item of submitted evidence, an analyst must determine the homogeneity of the samples. Once the number of populations is determined, a sampling plan can be employed to do presumptive and confirmatory testing.

- If the item is plant material, the analyst must be aware of state weight thresholds. Any units within the item that do not conform to the analyst's homogeneity expectations must receive a separate analysis according to protocols.
- If the item is not plant material (powder, tablets, blotter squares, etc.), the analyst must determine visually how many populations are within the item, segregate the units within the item, and employ sampling protocols in the selection of items for analysis. See Section 2.9 for sampling protocols. (Example: If an item contains 25 baggies of white crystalline substance and 3 baggies of off-white powder, the analyst would first segregate the items into two groups (A and B) before sampling for analysis. All testing is recorded in the analyst's bench notes.)

2.2 LITERATURE REFERENCE

Identification by a literature reference refers to:

- The identification of pharmaceuticals in dosage unit form utilizing shape, color, and manufacturer's markings/imprints. Literature reference of pharmaceuticals is appropriate when:
 - a. a presumptive physical identification of a product or ingredients is sufficient before reporting a non-controlled substance, or
 - b. triaging of multiple items in a case has identified the pharmaceuticals as not apparently vital to the total prosecution, or
 - c. physical identification is done as a secondary test to chemical analysis when an absolute identification is required.

Literature reference searches on pharmaceuticals (tablets, pills, capsules) are *inappropriate* when:

- a. it is the only test and a conclusive identification is required,
 - b. counterfeiting or mislabeling is suspected, or
 - c. an illicit pharmaceutical is involved.
- Literature search also can refer to the identification of drugs that have been found in instrumental data by use of a reputable spectral search library, but no standard is available in the laboratory for confirmation (GC/MS) or standard protocol utilizes a search function library (FTIR).

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

- Physician's Desk Reference (PDR)TM, Medical Economics
- Logo IndexTM, DEA
- IdentidexTM, Micromedex Inc.
- Drug Identification BibleTM, Amara-Chem, Inc.
- Government and manufacturer's websites (A printed image of the pharmaceutical is required when using a manufacturer as a reference.)
- Ident-A-DrugTM, Therapeutic Research Center
- Pharmer.org, Drugs.com, RxList.com, or other information websites
- Direct information from a pharmacist or manufacturer (Notes must indicate the source of the information.)

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2.3 QUANTITY DETERMINATION

Mass shall be determined in metric units on top-loading balances. Actual balance readings are recorded in the analyst's notes and not rounded or truncated in those notes.

A net weight will be obtained on all powders, plant material, and physical substances 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 may be weighed or a volume measurement obtained. Large seizures of liquid evidence may be estimated.

Tablets or capsules will receive a count or weight.

The method of measurement, either net or gross, shall be indicated in the notes. All weights in the report are net weights unless otherwise indicated.

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.
- For liquids, volume or weight may be reported. When weights or volumes are estimated, that information is communicated in the report.

For reporting purposes, weights will routinely be truncated to one decimal point (tenth 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".

If a count of pharmaceuticals, blotter squares, or individual packages differs from what the submitting agency has recorded on the RLS, a second laboratory person shall perform a count; this person need not be competency tested in the Chemistry discipline. The verification of the count should be recorded in the notes.

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2.4 COLOR TESTS

The following color tests are conducted as described below. For preparation of reagents, see Section 5 REAGENT PROCEDURES. The actual amount of sample needed for a color test is dependent on the concentration of the drug and the sensitivity of the test to the drug in question. The analyst should use a minimal amount of material.

Color tests do not provide structural information and as such are considered presumptive for the presence of a substance.

Reagents are tested at the time of preparation to ensure that they are functioning properly, and the results are recorded in the Reagent Log Book. All reagents will be routinely re-tested every year and the results recorded in the Reagent Log Book.

Commercially prepared ("NIK Kits") test kits may be used in place of laboratory prepared reagents. When doing so, one kit from each lot number will be tested and recorded in the Reagent Log Book. Manufacturer's supplied instructions will be followed. The lot number of the reagent shall be recorded in the case notes.

MARQUIS TEST

Place 1-3 drops of Marquis Reagent in a clean white spot plate or test tube and add several particles (or dried liquid residue) of the sample; observe and record response. Opiates give a characteristic violet color, while amphetamines give a red/orange. Do not confuse legitimate color reactions with charring reactions from the sulfuric acid. Be careful to distinguish the red/orange color of amphetamines from the gold or dull brown color that sometimes occurs with ephedrine. Note that reactions are faster and more intense with fresh reagent.

violet
gray to violet-black
yellow
orange to brown
slow pink to rose
yellow

opiates (heroin, morphine, codeine, etc.)
MDA, MDMA, propoxyphene
diphenhydramine
amphetamine, methamphetamine, phentermine, mescaline
aspirin
methylone (3,4-methylenedioxymethcathinone)

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SCOTT'S TEST (Cobalt Thiocyanate)

Place sample in a small tube. Add approximately 5 drops of Scott's Reagent to sample and shake. Observe for the formation of blue color and/or precipitate. Cocaine salts give a clumpy blue precipitate while cocaine base gives no reaction at this step. Follow with the addition of approximately one drop of concentrated HCl and observe blue color disappear and the color turn to pink. Add several drops of dichloromethane and shake. The dichloromethane (lower) layer will develop an intense blue color if cocaine (salt or base) is present. Cocaine salt gives a positive test, which is blue/colorless or pink/blue dichloromethane.

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 cobalt thiocyanate solution. Since the acid converts base cocaine to salt, both forms will give a positive reaction to the first step of this test.

WEBER TEST (For Psilocyn in mushrooms)

Add a small 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. Use a blank well or negative mushroom sample for a negative control. Add Weber Reagent and look for a purplish-red color. Then add one drop of concentrated HCl. A navy blue color indicates psilocyn.

PARA-DIMETHYLAMINO BENZALDEHYDE TEST (p-DAB or Van Urk's)

Although this reagent has its primary use as a thin layer chromatography color developing chemical for hallucinogens, it can also be used as a spot test for procaine and is reported to react with some synthetic cannabinoids in a procedure involving a methanol extract and heat.

Yellow
Violet

procaine
LSD, psilocyn, psilocybin

STARCH TEST (for Iodine)

Iodides may be identified by means of a well-known color reaction in which free I₂ gives a blue color with starch. This may be accomplished by utilizing:

- a test tube of suspected iodine with a cotton plug which has been soaked in starch solution, or

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- a white spot plate with suspected iodine in a well, covered with a microscope slide smudged with starch.

Gentle heating may speed the reaction. If item is an iodine tincture (iodide), addition of an oxidizing agent will convert the iodide I^- to iodine I_2 .

References:

- Moffat, A.C., *Isolation and Identification of Drugs*, 2nd Edition, edited by E.G.C. Clarke, The Pharmaceutical Press: London, 1986, pp. 160-177, 1170-1171.
- Butler, W.P. "Methods of Analysis for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs", Internal Revenue Service Publication No. 341, Washington, D.C., 1967, pp.92-93.
- Moffat, A.C., Osselton, MD, and Widdop, B, *Clarke's Analysis of Drugs and Poisons*, 3rd Edition, Pharmaceutical Press: London, 2004.
- Gardner, Vickie, *Memorandum: Presumptive Tests to Identify "Bath Salts"*, FDLE Criminal Investigations and Forensic Science Program, February 1, 2011.

2.5 CRYSTAL TESTS

The following crystal tests are conducted as described below. For preparation of reagents, see Section 5 REAGENT PROCEDURES. Reagents are tested at the time of preparation to ensure that they are functioning properly, and the results are recorded in the Reagent Log Book. All reagents will be routinely re-tested every year and the results recorded in the Reagent Log Book.

Crystal tests are performed by placing a minute amount of the sample on a microscope slide (in some cases the sample must be dissolved in a solution such as dilute acid) and adding a drop of the reagent directly to it. A second technique is to slowly bring two solutions together with a glass rod or applicator stick. Crystals are then observed under a polarizing microscope.

Criteria for a POSITIVE response:

- Test droplets that have dried may not be used for evaluation of crystals.
- Crystals must be compared to those of standards tested in the same manner.
- For frequently observed crystals, standards need not be run each time samples are run.

Gold Chloride Crystal Test for Cocaine

Reagents: 5% $HAuCl_4 \cdot 3H_2O$ in distilled water
20% Acetic Acid

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Procedure: Place sample on a glass slide and add 1-2 drops acetic acid. Add one small drop of gold chloride solution and observe immediately on the polarizing microscope for the appearance of long rods with one or many arms at nearly right angles to the main axis (+’s).

Reference: Butler, W.P. “Methods of Analyses for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs”; Internal Revenue Service Publication No. 341: Washington, D.C., 1967.

Silver Nitrate / Cupric Nitrate Crystal Test for GHB

Reagent: 100 mg of AgNO_3 and 100 mg of $\text{Cu}(\text{NO}_3)_2$ dissolved in 10 mL of water

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

If suspect material is a solution, combine a drop of unknown with a drop of reagent via a connection made by an applicator stick.

Reference: Andera, Kevin, 93rd Semi-Annual CAC Seminar, Oakland, CA; May, 1999

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2.6 PROTOCOL for MARIJUANA and MARIJUANA PRODUCTS

The protocol for marijuana or Cannabis analysis is dependent upon the form submitted. When plant material is present, the identification will include both a botanical and a chemical identification.

The entire sample should be visually examined for homogeneity. Generally, marijuana products can be divided into:

- Plant material, crushed plant material, charred plant material, or seeds are all controlled as VIA if seeds are viable and charred material includes identifiable plant hairs (reference statute definition):
- Residues, hashish, hash oil, and synthetically-produced cannabinoids (dronabinol or nabilone) are all controlled as IIIA (reference statute definition):

Macroscopic examination:

Record a description of the item and packaging in the notes. Indicate the results of the morphology examination. 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

Stereomicroscopic examination:

For plant material, observe sample for hairs on leaf material. Upper side of leaf bears unicellular claw-shaped cystolithic hairs containing CaCO_3 deposits at base. Some cystolithic hairs may be short with an enlarged base, giving a warty appearance. The underside of the leaf bears covering hairs that are longer and thinner than the opposing cystolithic hairs, and have no cystolithic deposits. Glandular hairs may be found on either surface of the leaf. These may appear in globular or mushroom-shaped form. Glandular hairs may appear wet and glistening due to presence of resin exuded by them. Glandular hairs are most profuse on flowering tops. Dried leaves tend to curl, with edges curling toward the lower surface.

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For hashish preparations, a microscopic examination should distinguish between ground-up marijuana and true hashish, which is primarily plant hairs. Identification of characteristic plant hairs combined with a chemical identification is sufficient for the identification of hashish.

Hash oil may be green or brown and will have no cellular material.

Note that a botanical identification is not usually possible with residue amounts.

Germination of seeds to demonstrate viability is not normally performed in this laboratory. Approval by the discipline supervisor or Laboratory Manager is required for this specialized testing.

Chemical Identification:

Extract a portion of the plant material or swab of suspected substance with a suitable solvent:

- Borate buffer/dichloromethane
- Petroleum ether
- Methanol

If a residue only is being examined, two samplings should be obtained, extracted, and analyzed.

Analyze by GC/MS. The minimum criteria for chemical identification of marijuana or marijuana products (THC, hash, hash oil, Marinol™, etc.) is the presence of tetrahydrocannabinol (Δ -9 THC).

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2.7 PROTOCOL for GENERAL DRUG (OR UNKNOWN) SUBSTANCES

The analysis of unknown samples can be accomplished by a variety of methods. The following is a generic protocol that outlines the analysis of a suspected drug sample. It should be noted that the nature of the sample determines the analytical route the analyst pursues and that many samples do not lend themselves to an exact order of protocol. The forensic scientist ultimately decides the specific route for each sample. Analysts have discretion on which samples will be processed.

Review the RLS form for latent fingerprint requests. If latent print work has been requested, special care in handling is required to prevent damage to any latent prints which may be present.

Repackage analyzed items that also require latent print analysis. If desired, digital imaging may be employed to document the original condition of the item. Perform a preliminary examination of the item. If one substance is present, proceed to examine the item. If more than one "population" appears to be present, or multiple packages are present, follow Section 2.9 Sampling Protocols.

- Record a description of the item and packaging in the notes.
- Obtain a weight, count or volume of sample.
- Perform any applicable preliminary testing on sample.
- Prepare sample for analysis.
- Analyze the item.
- The analyst may do more testing than the minimum required.
- Unapproved tests, if used, shall not be counted as part of a minimum analytic scheme.

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2.8 PROTOCOL for CLANDESTINE LABORATORY EVIDENCE

The objective in analyzing clandestine laboratory evidence is to determine if either a suspected lab site has the capacity to manufacture methamphetamine or has in fact done so. The ephedrine/pseudoephedrine reduction method is the method most commonly observed. Precursors and chemicals include:

Ephedrine or pseudoephedrine

This precursor is normally extracted from over-the-counter tablets.

Iodine

This reagent may be seen as crystals of I_2 or as solutions of iodide, I^- . Rarely, labs may have access to HI (hydroiodic acid), but the usual source of HI is the combination of iodine and any acid or iodine and phosphorus.

Phosphorus

Red phosphorus is a reddish-brown powder. It may be obtained from chemical supply houses, road flares, fireworks, and matchbook striker plates.

The other type of ephedrine reduction utilizes lithium (or sodium) metal and anhydrous ammonia:

Lithium

Lithium is extracted from lithium batteries with tin snips or obtained commercially. It is usually stored in toluene or mineral oil because of its explosive, reactive nature with water. *Lithium is not analyzed in this laboratory.*

Sodium

Obtained from a chemical supply house or made by electrolytic deposition, using sodium hydroxide, copper tubing, and a car battery. Sodium metal reacts violently with water, causing fire and explosion. *Sodium is not analyzed in this laboratory.*

Ammonia gas

This may be found stored in pressurized cylinders or propane tanks, often in agricultural settings (as fertilizer), fish-processing plants, and ice-rinks. Gas

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is released into a thermos bottle as a liquid. *Ammonia is not analyzed in this laboratory.*

Other chemicals may be submitted as evidence but are not typically analyzed:

- **Acids** (Hydrochloric or Muriatic, Sulfuric)
- **Solvents** (Toluene, Coleman fuel, Acetone)
- **Alcohols** (for pill extraction)
- **Drying Agents** (Epsom salt)
- **Filters** with Residues
- **Tubing** from HCl gas generators
- **NaOH**, usually as a liquid drain cleaner

Filters or tubing with drug residue may sometimes be analyzed for finished product.

PHOSPHORUS by GC/MS

Phosphorus can be identified by GC/MS after converting red phosphorus to white phosphorus.

A small amount (1-2 mg) of red/brown powder substance is added to a test tube. The tube is then heated in the flame of a propane torch. Perform this procedure under a fume hood and always wear safety goggles! As the red phosphorus burns, white phosphorus begins to accumulate on the sides of the test tube. Remove the tube from the flame after 10-15 seconds, allow to cool, and add 1-2 milliliters of dichloromethane. Be extremely careful of flash back at this point, as the dichloromethane is volatile and the phosphorus is explosive. Transfer the dichloromethane/white phosphorus mixture to an auto sampler vial.

Inject the sample on the GC/MS. Once the experiment is complete, look for a peak with the ions (mass/charge) of 31 (P1), 62 (P2), 93 (P3), and 124 (P4). Compare unknown spectra to that of a phosphorus standard prepared in the same way as the unknown sample.

IODINE

Iodides may be identified by means of the well-known color reaction in which free I_2 gives a blue color with starch. See **Section 2.4 Color Tests**.

Reference: Chamot and Mason, Handbook of Chemical Microscopy, Volume II

Distillation in the well of a spot plate (or in a test tube) is a means of separating the liberated I_2 from substances that might interfere with the "starch-iodide" test. A small amount of the substance to be tested is placed in the well of a spot plate or a test tube. Several drops of dilute acid containing an oxidizing agent may be added to oxidize any I^- to I_2 . Cautious heating aids in volatilizing the I_2 . A few granules of starch placed on a microscope slide or a

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cotton ball are placed over the well or in the test tube respectively. A blue color indicates the presence of iodine.

Sublimation can also be considered as indicative of iodine. Blue-black iodine crystals can convert directly from solid form to a violet-colored gas phase.

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2.9 SAMPLING PROTOCOLS

The following guidelines dictate the laboratory's policies on sampling. Conclusions reported must clearly state to the user of the report exactly what was analyzed to reach the stated conclusions.

- General sampling guidelines:
 - Visually different samples within an item will be separated for analysis.
 - Visually different samples within an item with potential cross contamination may not need separate analyses.
 - The analyst should leave a portion of the original sample for potential re-analysis.
 - A minimum of two samplings shall be separated for analysis from each item to be tested (one presumptive and one confirmatory).
- Single sample items:
 - If the sample appears homogeneous, a portion is simply removed.
 - If an item is not homogeneous (for instance, cocaine salt mixed with cocaine base) or is very large, multiple sampling can be done. The worksheet and report should reflect the mixture of drug materials.
- Multiple sample items
(Before employing sampling protocols, be familiar with Section 2.1 Visual Screening and Appendix II Weight Considerations in the Alaska Statutes):
 - If the gross weight of the entire population is above a critical weight and 20 or less samples from that population are required to exceed that critical weight, full analysis will be performed on that number of samples regardless of population size.
 - If the gross weight of the entire population is above a critical weight and more than 20 samples from that population are required to exceed that critical weight, a hypergeometric sampling plan (90% positive with a 95% confidence interval) will be used to determine the number of samples to be analyzed (see Appendix IV Hypergeometric Sampling Plan). If any samples differ from the rest, the analyst will reevaluate the item population.
 - If the gross weight of the entire population is below the minimum critical weight or there are no critical weights for the drug in question, only one sample may be analyzed.

Example A: 55 tablets, all appear visually the same with pharmaceutical markings

- Record description of packaging and tablets in notes.
- Weight or count of all tablets is recorded in notes.
- Literature search (unless clandestine)

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- If controlled, perform GC/MS of one tablet
- If not controlled, report drug and “physical identification only”
- **DESCRIPTION:** Blue tablets
55 count
1 tablet analyzed
- **RESULTS:** Oxycodone, 1 tablet

Example B: 8 baggies of plant material, all appear visually the same

- Record description of packaging and contents.
- Perform a gross weight of all 8 baggies.
- If the total weight apparently exceeds a threshold amount of 1 ounce or 4 ounces, perform a botanical identification and GC/MS on as many baggies as needed to reach the threshold amount.
- If the total gross weight is less than 1 ounce (28 grams), perform a botanical identification and GC/MS on the contents of *one* baggie & repackage.
- **DESCRIPTION:** 8 baggies Plant Material
Gross weight, xx.x grams
Contents of 1 baggie analyzed
- **RESULTS:** Marijuana, x.x grams

At an analyst's discretion, additional presumptive or confirmatory testing may be performed beyond the sampling plan policy.

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2.10 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. The choice of an organic solvent is dependent upon the drug to be extracted and the preferences of the analyst.

- Solvent extraction - the analyte is dissolved in an appropriate solvent and separated away from insoluble excipients. The solvent is then analyzed.
- Solvent washes - the excipient material is dissolved and washed away from the analyte by using a solvent in which the analyte is insoluble.
- Solvent/solvent extractions - two immiscible solvents can be used to extract an analyte. An aqueous phase (acidic, basic, or neutral) and organic solvent phase are typically used. Many drugs are nitrogenous compounds that readily convert between salt and free species enabling them to be separated by acid/base and organic solvents.
- Particle-picking - some mixtures and crystalline samples can be physically separated and isolated. A stereomicroscope can aid in the isolation of individual components of a sample.

Physical Separation:

Manually separate material by physical appearance. This includes scraping or shaking material from currency, paraphernalia, clothing, etc.

Dry Extraction:

- Place a portion of the sample in a disposable test tube.
- Add a solvent that dissolves the drug of interest, but not excipients.
- Decant or filter to separate the solvent and discard any insoluble material.

Collection of Residues

Paraphernalia may be rinsed by pouring solvent onto/through the item OR swabbing with a cotton-tipped applicator moistened with water or solvent.

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Liquid/liquid extraction:

- Basic extraction
 - Dissolve the sample in ~ 5% borate buffer (or other suitable basic solution).
 - Add dichloromethane (or other appropriate solvent).
 - Vortex or shake.
 - Resulting layers contain:
 - Aqueous Layer – Acidic chemicals.
 - Organic Layer – Basic and neutral chemicals.
- Acid extraction
 - Dissolve the sample in 0.1N H₂SO₄ (or other suitable acidic solution).
 - Add dichloromethane (or other appropriate solvent).
 - Vortex or shake.
 - Resulting layers contain:
 - Aqueous Layer – Basic chemicals.
 - Organic Layer – Acidic and neutral chemicals.
- Acid/base extractions (Back Extractions)
 - Dissolve sample as described above.
 - Add organic solvent as described above.
 - Shake and separate layers.
 - Resulting layers contain:
 - Acidic Aqueous Layer - Basic chemicals.
 - Adjust pH of aqueous layer to ~ 9-14 using base.
 - Add dichloromethane.
 - Shake.
 - Resulting layers contain:
 - Basic Aqueous Layer - Organic insolubles.
 - Basic Organic Layer - Basic chemicals
 - Acidic Organic Layer - Acidic chemicals.

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

- Combine approximately equal volumes of the sample liquid and dichloromethane in a test tube. (2 – 3 milliliters of each when sample size permits). If the sample is powder, take up in water, and then extract with dichloromethane.
- Vortex or shake.
- Let settle.
- GBL/BD will be in the dichloromethane layer. If Gamma-hydroxybutyric acid (GHB) is present, it will be in the aqueous layer. Remove dichloromethane and analyze by GC/MS.

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Gammahydroxybutyric Acid (GHB) Procedure: Perform a microcrystalline test and then if positive proceed with the following protocol:

- Remove a portion of sample (or take the aqueous layer from "E" above) and evaporate to dryness. Keep dried sample at ~105° C or place in a dessicator.
- GHB Derivatization Procedure using N-methyl-N-trimethylsilyltrifluoroacetamide (MSTFA)
 - Place MSTFA in three auto-sampler vials or auto-sampler vials with glass inserts.
 - Add a couple of mg of 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 MSTFA.
 - Derivatize capped vials at 90° C for 10 minutes (hotplate or warm water bath).

Mushroom Extraction Procedure:

- Grind mushrooms to a fine powder.
- Soak 1 to 2 grams in ethanol for 30 minutes, vortexing every 15 minutes for 30 seconds.
- Add 10 drops of 20% acetic acid.
- Soak an additional 30 minutes with vortexing for 30 seconds.
- Centrifuge sediment to bottom and transfer acidic ethanol to a test tube.
- Evaporate to dryness.
- Reconstitute with dichloromethane, 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 milliliters of 15N ammonium hydroxide.
 - Stir the mixture.
 - 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 milliliters of dichloromethane and methanol mixed in a 3:1 ratio.
 - Mix vigorously.
 - Centrifuge or allow to settle.
 - Remove the liquid and filter it through a filter presoaked with the dichloromethane:methanol mixture.
 - Add 1.5 to 2 milliliters of water to the filtrate and mix thoroughly.

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- 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

LITERATURE REFERENCES

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- H. Timmons, J.E., The Identification of Psilocin and Psilocybin Using Gas Chromatography-Mass Spectrometry, Microgram, Vol XVII, No. 2, February 1984.

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2.11 NEW METHOD VALIDATION

This procedure will apply to new method validations used by the analysts in the controlled substances discipline. Actual validation protocols will vary, depending upon whether the method is laboratory-developed or has been already validated at another certified laboratory.

- Perform a literature search in the area of interest.
- Develop specific objectives for the method. Include reasons why the procedure may be of value. Discuss development of the method with supervisor and other discipline members.
- Plan a series of steps required to perform the experiment (methodology).
- Perform the experiment. Note any changes made to the initial procedure.
- Prepare a validation/verification report documenting the study. Subjects addressed in the report should include:
 - Objective
 - Theory/background
 - Chemicals/reagents/equipment
 - Instruments/parameters
 - Methodology/procedure
 - Summary of data/results
 - Spectra/chromatographs/tables/graphs
 - Discussion of results
 - Applicability
 - Limitations
 - Specificity
 - Reproducibility
 - Usefulness
 - Bibliography of references and cited works
- Submit report to the Controlled Substances Supervisor for approval.

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SECTION 3 INSTRUMENTAL PROCEDURES and QUALITY CONTROL

3.1 GAS CHROMATOGRAPHY / MASS SPECTROMETRY

The State of Alaska Scientific Crime Detection Laboratory utilizes GC/MS instruments for analysis of drug substances. Information regarding all instrument specifications is kept:

- In individual instrument logbooks kept with the instruments in the Instrument Room. This includes specific methods and operating parameters,
- I:\Discipline Shares\Controlled Substances\Instrumentation
- In the LIMS under CS INST 20XX (Where XX is the year):
 - Unique identifier
 - Serial numbers
 - Software version
 - Manufacturer
 - Location, and
- Through the instrument software HELP function, which accesses the electronic instrument manual and gives specific instructions on the use of the GC/MS and software
- Manufacturer's electronic hardware manual for GC/MSD.

Limitations: Not all samples are amenable to GC/MS analysis. Thermally labile and non-volatile samples are not readily analyzed.

- Complete destruction occurs with the injected portion of evidence.
- Spectra from closely related compounds and isomers may be so similar that conclusive identification is not possible.

Procedure for GC/MS analysis:

- Prepare blanks (negative controls) to precede each casework sample being analyzed. Run a blank before each evidence sample using the same instrument conditions (oven temperature program, split ratio, injection volume, & multiplier voltage). The purpose of the negative control is to demonstrate a lack of contamination in both the solvents used to prepare a sample, and the instrument.
- Prepare sample solutions according to *Section 2.10 Extractions* and transfer to autosampler vials.
- Make sure the liquid level is above the syringe injection depth.
- A temperature program encompassing the range of the Quality Assurance Mixture must be used to assure resolution of drug components.

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- Sample injection shall be by autosampler.
- The following information shall appear on the printout header:
 - The GC/MS laboratory identifier.
 - The GC/MS laboratory identifier.
 - Acquisition program utilized (Screening). Details of the program method are referenced in each instrument's Log Book.
 - Laboratory case number
 - Item number.
 - Analyst
 - Date and time of sample injection
- A standard must be referenced for each substance reported and included in the case file. SCDL verified standards shall be the first choice of comparison, but literature standards will be used if a SCDL standard is not available.
- When instrumental acquisitions are complete, the analyst will review the data and put a copy into the case file. Each electronic copy will include the header information listed above, a total ion chromatogram, and the mass spectrum of each peak investigated.
- 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.
 - When instrumental acquisitions are complete, the analyst will review the data and put a copy into the case file. Each electronic copy will include the header information listed above, a total ion chromatogram, and the mass spectrum of each peak investigated.
 - 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.

Interpretation of Data and Criteria for Identification by GC/MS:

Blanks must not contain peaks of interest. Documentation of the blank samples is retained in the case file.

All spectra from analyzed substances will be compared to spectra from verified in-house drug standards, when available. Documentation of these standards is retained in the case file. If a chemical standard is not available, an identification can be made utilizing approved spectral libraries or approved outside sources.

The mass spectrum of each sample is visually compared with that of a primary standard. 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 main ions should agree between unknown and standard and the presence or absence of a 'molecular ion' must agree between unknown and standard. Unknown sample spectra may

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contain additional peaks due to background or sample impurities. If the spectrum from an evidence sample is lacking in detail and the analyst feels that it is too weak to call, the analyst may report "insufficient quantity for complete identification". This will indicate that the analyst did perform an analysis, sufficient data existed to suggest the presence of a specific substance, but the results did not meet the criteria for identification. The criteria for the acceptance of a spectral match between unknown evidence and a known standard is the same whether the standard is from a reputable library or analyzed in-house from the laboratory's drug inventory.

The mass spectrum of each sample is visually compared with that of a primary standard. 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 main ions should agree between unknown and standard and the presence or absence of a 'molecular ion' must agree between unknown and standard. Unknown sample spectra may contain additional peaks due to background or sample impurities. If the spectrum from an evidence sample is lacking in detail and the analyst feels that it is too weak to call, the analyst may report "insufficient quantity for complete identification". This will indicate that the analyst did perform an analysis, sufficient data existed to suggest the presence of a specific substance, but the results did not meet the criteria for identification. The criteria for the acceptance of a spectral match between unknown evidence and a known standard is the same whether the standard is from a reputable library or analyzed in-house from the laboratory's drug inventory.

The following are accepted as searchable electronic library references for use in establishing the identification of drugs found by GC/MS when the laboratory lacks a chemical standard:

- Agilent "Identification of Synthetic Cannabinoids by GC/MS"
- SWGDRUG
- DEA
- NIST
- Wiley
- Alaska Scientific Crime Detection Laboratory in-house library (DRUG)

The following are accepted as non-electronic library references for use in establishing the identification of drugs found by GC/MS when the laboratory lacks a chemical standard:

- CLIC
- DEA MicroGram
- FBI Laboratory
- DEA Research and Testing Laboratory
- Other citations as approved

As with any instrumental analysis, the notes shall indicate that GC/MS was performed, what extraction was done, and what results were found.

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Uncertainty of Measurement of GC/MS and Good Lab Practices

The biggest uncertainty in detection and measurement when utilizing GC/MS technology is contamination prevention. The second potential source of error is in mislabeling or misplacement. To assure that contamination or mishandling of evidence does not occur, the laboratory adheres to the following practices:

- Only one item of evidence should be analyzed at a time.
- Use disposable gloves, bench paper, pipets, wipes, test tubes, weigh papers, and vials when handling evidence.
- Reusable items (spatulas, forceps, beakers, scissors, etc.) must be cleaned prior to each use.
- Balances should be cleaned as needed and used with disposable trays.
- Batch processes (autosampling) require that each item be labeled.
- A reagent blank or solvent blank should be prepared and run on the GC/MS prior to each case sample. Documentation of this is maintained in the case file.
- If an extract is to be concentrated using the water bath evaporation system, the probes are cleaned prior to use.

Maintenance of GC/MS instruments

The following protocols are established as a preventive maintenance program for gas chromatograph/mass spectrometers. All maintenance, both preventive and corrective, must be described and dated when recorded in the instrument logbooks. Exceptions to this include routine computer re-booting and exchange of helium tanks.

The SCDL maintains service contracts with Agilent for technical support, service, and parts. At this time, the service contracts do not include any routine preventative maintenance (source cleaning, pump oil changes, adjustments, lubrication, inspections, or column changes), so these actions are normally performed by crime lab personnel on site. On occasion, analysts may be called upon to install filaments, a voltage multiplier, or other replacement parts.

Columns that are installed in the Gas Chromatograph/Mass Spectrometer shall have an approximate length of 30 meters and have a 5% phenyl/95% polymethyl silicone phase, 100% methyl silicone, or an alternate column as approved by the discipline supervisor.

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Maintenance cuts to the columns to enhance column performance are permitted. These columns will be used to run the QC mix and for general drug analysis. Columns are replaced as needed.

Compressed helium is used as a carrier gas. Tanks are stored in the building and the gas is transported to the instruments in metal tubing. A manifold system allows rapid change from one tank to another when the gas is nearing depletion. New tanks of helium are obtained from Air Liquide, an ISO 17025 certified supplier. Installation of new tanks may be performed by the laboratory's maintenance specialist or anyone in the controlled substances discipline of the laboratory.

Septa, glass inlet liners, autosampler syringes, and other consumables are replaced as needed.

Quality Control of GC/MS:

Perfluorotributylamine (PFTBA)— calibration standard. The GC/MS instruments are calibrated weekly (when in use) utilizing the Autotune program. Reports of calibrations (Autotune reports) are electronically stored in the Laboratory Information Management System (LIMS) in a file designated **CS INST 20XX** (where XX is the current year).

A successful calibration is indicated when the instrument is able to assign proper masses to the PFTBA fragments. Critical components are:

- Passing the System Verification in the Tune Evaluation program
- Peaks are symmetrical in shape.
- Mass 69 is the base ion.
- Water and air abundances pass the Tune Evaluation (are less than 20% of the 69 abundance).

Quality Control (QC) Mix: A mixture of four 1 mg/mL drug substances specifically selected to demonstrate the retention time range for controlled substances in Alaska and definitive peak resolution between closely related drugs :

- Dimethyl Sulfone (MSM)
- Tetracaine
- Cocaine
- Buprenorphine

The QC Mix is analyzed weekly on each instrument. Each GC/MS must identify each of the four components with acceptable GC resolution, sensitivity, and peak shape on the total ion

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chromatogram (TIC) and acceptable mass spectra for the cocaine peak. A successful performance check is indicated by:

- Detection of the four components of the QC Mixture.
- Baseline resolution between tetracaine and cocaine.
- Symmetrical peaks with minimal tailing.
- The cocaine peak of the QC Mix giving an appropriately detailed mass spectrum when compared to a reference standard spectrum.

Each GC/MS must pass QC check (autotune calibration and QC Mix control) each week before it can be used in casework. Any maintenance such as a column change or hardware replacement requires a QC check before casework can be resumed with that instrument. An autotune is recommended after liner and septum changes but not required.

QC Evaluation: If any of the criteria above are not met, then the instrument does not pass the quality control check and must not be used in casework. The instrument must undergo further maintenance and/or troubleshooting to correct any problems and a subsequent quality control check must be performed. Entry of Autotune, Tune Evaluation, and QC Mix data into the electronic data storage of Justice Trax is made when the data satisfies all requirements.

Records:

The following documents must be maintained in the laboratory for each GC/MS. Some data may be duplicated between hard copy in the logbooks and electronic storage in LIMS:

- A description of the instrument system
- Documentation of weekly calibration (Autotune) data.
- Documentation of weekly QC Mix data.
- Documentation of all maintenance and repairs.

Bibliography

Saferstein, R., Ed. *Forensic Science Handbook*; Prentice-Hall: Englewood Cliffs, NJ, 1988, Vol.III.

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3.2 INFRARED SPECTROPHOTOMETRY (FTIR)

The State of Alaska Scientific Crime Detection Laboratory utilizes a Thermo Nicolet iS10 Infrared FTIR Spectrometer with an ATR (attenuated total reflectance) accessory for analysis of drug substances. Information regarding the instrument specifications is kept in the instrument logbook and manuals kept with the instrument in the Instrument Room. Information on the infrared spectrometer is also stored on the Lab's computer network: <I:\Discipline Shares\Controlled Substances\Instrumentation\FTIR>.

Infrared analysis is non-destructive and sample size is small. However, the sample must be relatively pure for conclusive identification. Inorganic compounds often lack the spectral complexity necessary for identification.

Procedures for FTIR analysis:

- 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.
- Acquire a background spectrum (blank) of air. The blank must be analyzed using the same instrumental conditions as the sample and retained in the case file.
- Perform a quality control check by acquiring an absorbance spectrum using a 1.5 mil film of polystyrene.
- Place small amount of sample on ATR crystal.
- Acquire sample absorbance spectrum and search libraries for an identification

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.

The following are accepted as references for use in establishing the identification of drugs found by FTIR:

HR Georgia State Forensic Drugs
Alaska Scientific Crime Detection Laboratory library

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Maintenance:

- Record all maintenance in the instrument logbook kept beside the FTIR in the instrument room.
- Routine maintenance performed as needed:
 - Dessicant replacement
 - Inspection of humidity indicator
 - Cleaning of mirrors (factory service only)
 - Alignment of spectrometer
 - Cleaning of spectrometer, accessories, computer, monitor, and keyboard

Quality Control:

- Perform a background acquisition on all days that the instrument is used.
- Acquire spectra from a polystyrene standard. Check for absorption at 1601 cm^{-1} , $\pm 2\text{ cm}^{-1}$. This peak can be labeled using the 'T' function in the annotate toolbar. 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 insert the spectra into the LIMS file.
- Instrument validations are run after any major service or hardware replacement. The manufacturer's software program shall 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 all validations (performance verifications) in the LIMS in the folder CS INST 20XX.

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

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

References

Smith, B.C. *Fundamentals of Fourier Transform Infrared Spectroscopy*; CRC Press: Florida, 1996.

Karasiewski, R. *Basic Training Program for Forensic Drug Chemists*; 3rd Edition, Drug Enforcement Administration, 1998, Chapter 5, pp. 29-48.

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3.3 BALANCES

The State of Alaska Scientific Crime Detection Laboratory utilizes balances for weighing of substances. Information regarding the balance specifications is kept in the Balances Logbook and the manuals stored in the Controlled Substances laboratory. Additionally, this information is stored on the Lab's computer network: [I:\Discipline Shares\Controlled Substances\Instrumentation\Balances](#) and in the LIMS folder CS INST 20XX.

Maintenance

- Make sure the balance is level and protected from vibration.
- Keep all balances clean and dust-free.

Quality Control

- All balances will be checked with NIST traceable standard weights on a monthly basis. Reference weights must not be touched with bare hands (tweezers or gloves are used).

XS2002S balances :	0.50 grams	±0.01 gram
	10.00 grams	±0.01 gram
	100.00 grams	±0.01 gram
MS16001L balances:	0.5 grams	±0.1 grams
	10.0 grams	±0.1 grams
	100.0 grams	±0.1 gram
	4000.0 grams	±0.1 gram

- When a balance is physically moved, it must be re-verified with the standard weights.
- All balance checks will be documented in the balance logbook.
- All new balances will be checked with NIST traceable weights prior to use in casework.
- All balances will be calibrated and serviced on an annual basis by a vendor utilizing
 - NIST traceable weights. The calibration will be recorded in the balances logbook and/or maintained in the Quality Assurance Manager's records.
- A repaired balance must be re-calibrated prior to use.

Uncertainty of Measurement Budget

- Operator Uncertainty – All personnel using balances in the testing of controlled substances are by training and experience fully qualified in the proper use of the equipment.
- Environmental Conditions – All balances are operated in a laboratory environment consistent with manufacturer's recommendations. Calibration checks and calibrations are performed at the locations where the balances are used.
- Calibration Procedures – All calibrations are performed in accordance with manufacturer's recommendations. Balances are checked monthly and re-calibrated if necessary. Annual calibrations are performed by an accredited vendor.

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- Weight (Mass) Standards – Proper handling procedures for these standards are documented in **Section 2.3 Quantity Determination**, **Section 3.3 Maintenance**, and **Section 3.3 Quality Control**. The standards have been certified as NIST traceable by the supplier (VWR/SP) and are re-certified on an annual basis by an accredited vendor. NIST certification documents are kept in the balance logbook and on the Laboratory computer network:
- I:\Discipline Shares\Controlled Substances\Instrumentation\Balances
- Instrument Performance – Instrument performance is checked monthly using NIST traceable mass standards. Measurements obtained are for a range of weights appropriate for each balance and relevant to laboratory needs. These measurements may potentially be used to calculate standard deviation values.
- Sampling – Since *net* weights of evidence are usually obtained, residual sample may be left in an evidence container (bundle, pipe, baggie, etc.). This difference in weight is not significant and will always result in a lower reported weight than what is actually present.
- Truncating – Policy requires that weight quantities be truncated. As with sampling (see above), any discrepancy is a lower reported weight than the actual weight.
- Moisture Content of Evidence – It is beyond the control of the laboratory to dictate the moisture content in submitted evidence. This can then potentially result in a change of evidence weight over time as the substance either loses or gains weight with the drying or absorbing of water from ambient air.

Records: The following documents must be maintained in the laboratory for the balances. Some data may be duplicated between hard copy in the Balances Logbook, hard copy kept with the Administrative Assistant and the Quality Assurance Manager, and electronic copies stored on the laboratory computer network:

- Descriptions of all balances with locations and serial numbers
- Documentation of monthly calibration checks
- Documentation of weight and balance validations
- Documentation of all maintenance and repairs

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3.4 INSTRUMENT INTERFACES (COMPUTERS)

Computers used to interface instrumentation in the laboratory shall be supplied by or approved by the manufacturer of the instrumentation.

Maintenance

Restart computers weekly (at a minimum) to activate upgrades and patches.

3.5 NEW INSTRUMENT VERIFICATION PROCEDURE

GC/MS: At this time, all installation and verification of new MSD's is done by an approved factory representative. The documentation of this process is kept in each instrument's log book and includes a checkout tune, tune evaluation, and sensitivity check. Also checked are PFTBA peak shapes, mass assignments, and mass axis stability. A signal to noise report and tune report are generated by the technician.

FTIR: New instruments or attachments to existing instruments will be verified by the factory representative and documentation provided to the Chemistry Supervisor or his/her designee.

Additionally, an in-house validation will be performed to demonstrate that the new instrumentation is equal or superior in performance to the replaced instrument.

A memo from the Controlled Substances Supervisor to the QA Manager documents a new instrument going into service, includes the serial number, and outlines the basis of the verification process. Paperwork with the memo includes the hardware and software installation checklist. All documentation included with the memo should ensure that the instrument was correctly installed and functioning as designed.

A memo from the Controlled Substances Supervisor to the QA Manager documents a new instrument going into service, includes the serial number, and outlines the basis of the verification process. Paperwork with the memo includes the hardware and software installation checklist. All documentation included with the memo should ensure that the instrument was correctly installed and functioning as designed.

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SECTION 4 ADMINISTRATIVE PROCEDURES

4.1 CASE FILE PROCEDURE

Bench Notes (Worksheet): The administrative aspects of casework analysis are found primarily within LIMS under the **Requests** for Analysis tab. It is here that the request is created, pertinent information added, prioritizations conveyed, evidence is requested, bench notes are recorded, reports are written, reviews are recorded, and distribution of the report is documented.

Proper note-taking is essential from the time the evidence is received by the analyst until returned to the Evidence department. Notes need to be complete and understandable by another discipline analyst, as they may be referred to months or years after the analysis was performed.

Proper notes should include the following information:

- Any inconsistency between the description on the submission form and the evidence received.
- Dates that the analyst began analysis and date completed. To imbed this information in the worksheet, go to **Edit Request** and enter the two dates in the Assignor block, using the format MM/DD/YYYY.
- Description of each item's packaging. Note any packaging problems or unusual circumstances.
- Physical description of the evidence (powder, liquid, plant material, tablet, color)
- Sampling protocol employed.
- Weight or count of item. If multiple units are present within an item, obtain a gross weight of the entire item before sampling protocols are employed. Record this weight on the worksheet.
- Sample preparations or extraction methods used.
- Description of each analysis and the result.
- Conclusions upon completion of analyses.
- All chromatograms and spectra generated by analysis.
- Reagent blanks.
- Standards.
- Literature references or monographs if needed.

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Case Images: This portion of the case file in LIMS contains all Requests for Laboratory Services (RLS's) and subpoenas associated with the case, as well as any additional case information submitted by the law enforcement agency or Department of Law. Items not already in electronic form that need to be saved with the file can be scanned and added to the LIMS images.

Communications: An acceptable form of documenting communications concerning a case is to use the Case Activities function (under the Case Info tab) in the LIMS database. Inquiries concerning specific aspects of a case such as triaging may be entered as a "customer query" by selecting Controlled Substances under CONTEXT Department, not selecting a Service, and then, under Activity, selecting "CS-Customerq". Tracking these communications allows the laboratory to document the need for responsive actions to our client base.

4.2 REPORTS

All results shall be entered into the approved laboratory computer format for report generation. The report that is issued represents a summary of the analytical findings and shall include:

- Name of submitting agency
- Submitting agency case number
- Date of report
- Crime Laboratory case number
- Name of submitting officer or contributor
- Brief description of items analyzed
- Weight, volume, or count of each item analyzed
- Description of any sampling protocols utilized
- Results, conclusions, and opinions
- Name and signature of the Forensic Scientist performing analysis
- Name of person performing technical review of findings

The final report will clearly convey to the officer and/or prosecutor exactly what was analyzed.

When an error is discovered in a report that has been reviewed and distributed, the discipline supervisor will be notified, the analyst and reviewer consulted, and a corrected report issued. If the error is serious enough to question the protocols, policies, or the judgment of the analyst, the Laboratory Quality Manager and Forensic Laboratory Manager will be included in any corrective actions.

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SECTION 5 REAGENT & STANDARD PROCEDURES

A reagent log book is used to document reagent preparation and verification. The log book will include:

- Reagent name.
- Date prepared.
- Assigned lot number with initials of person making reagent.
- Response to verification standards.
- Storage conditions and expiration date (if any).
- Initials of person verifying reagent.

Reagents may be re-tested with known positive and negative controls to assure sensitivity and absence of contamination if any problems arise or at the analyst's discretion. All reagents will be tested yearly and results recorded in the Reagent Log Book.

Marquis Reagent

20 mL concentrated sulfuric acid
16-20 drops formaldehyde (37%)

Expiration: 1 month

QA: Check with methamphetamine (orange) or an opiate drug (purple)

Reference: Clarke, E.G.C. *Isolation and Identification of Drugs*, 2nd edition; pp. 139-140.

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

Dissolve 1.0 gram of para-dimethylaminobenzaldehyde in 100 ml ethyl alcohol and 10.0 ml concentrated HCl.

Expiration: none

Comment: Although this reagent has its primary use as a thin layer chromatography color developing chemical for hallucinogens (ergot alkaloids), it can also be used as a spot test for procaine, psilocyn, and LSD.

yellow	procaine
violet	LSD, psilocyn, psilocybin

QA: Check with procaine or psilocyn

Reference: Clarke, E.C.G. *Isolation and Identification of Drugs*, 2nd edition; Pharmaceutical Press: London, 1986, p. 132.

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Scott's

Solution A: 2% cobaltous thiocyanate in water and then diluted 1:1 with 96% USP glycerine

Solution B: Concentrated HCl

Solution C: Dichloromethane

Expiration: none

Comments: The Scott's test is commercially manufactured into a standard field test for law enforcement officers (Scott Reagent Modified).

"Modified" Scott's reagent is prepared using 10% acetic acid instead of water. This reagent will give a positive reaction for salt or base in the first step.

References:

1. Scott, L.J., *Microgram*, vol.6, no.11 (November 1973) pp179-181.
2. Prall, J.D., *Microgram*, vol.8, no.9 (September, 1975) pp.130-132.
3. Fasanello, J. and P. Higgins, *Microgram*, vol.19, no.10 (October 1986)

Weber Reagent

Solution A: Add approximately 0.01 gram Fast Blue B Salt [o-dianisidine bis (diazotized) zinc double salt] to 10ml water. The solution will have a faint straw color.

Solution B: Concentrated HCl

Comments: The Fast Blue B reagent must be made *fresh* when the mushroom or mushroom extract is ready to be tested. Reagent is suitable for use only on the day it is prepared; at the end of the day the reagent will be appropriately discarded.

QA: Reagent must be tested when made with psilocin or known psilocin containing mushrooms. A red color change after the first step followed by a blue color change after the second step is a positive result.

References:

1. Lee, Henry & Harris, Howard, *Physical Evidence in Forensic Science*, 2nd Edition, Lawyers & Judges Publishing Co., 2006.
2. San Francisco Police Dept. Criminalistics Lab, *Controlled Substances SOP*, 2005, p.44.
3. Garrett, Clemens, and Gaskill, "The Weber Test: A Color Test for the Presence of Psilocin in Mushrooms", *SWAFS Journal*, Volume 15, Number 1, April, 1993, page 45.

Gold Chloride Reagent for Cocaine

5% HAuCl₄.3H₂O

20% Acetic Acid

Expiration: none

QA: Test with cocaine, hydrochloride or base.

Reference: Butler, W.P. "Methods of Analyses for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs"; Internal Revenue Service Publication No. 341: Washington, D.C., 1967.

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Silver Nitrate/Cupric Nitrate Microcrystal Test for GHB

100 mg AgNO_3 and 100 mg of $\text{Cu}(\text{NO}_3)_2$ dissolved in 10 mL of distilled or deionized water.

QA: Test with GHB primary standard and/or various concentrations of solutions kept in Toxicology freezer.

Expiration: none

Reference: Andera, Kevin, 93rd Semi-Annual CAC Seminar, Oakland, CA; May, 1999.

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5.2 STANDARDS

Primary Standards are controlled substances purchased from an approved provider (see VENDORS in LIMS with approved suppliers) or supplied by the DEA or an ISO-accredited crime laboratory. A copy of the current DEA license for purchase of controlled substances is found in a binder kept by the Controlled Substances Supervisor and is also stored electronically on the lab network. Records of the purchase of Schedule I and II (federal) drugs are also stored in the binder kept by the Controlled Substances Supervisor. Primary Standards may be a pure (neat) compound or a solution of a neat compound. Any certification documentation accompanying these standards is maintained in a binder in the controlled substances laboratory, but all drug standards must be verified prior to use. Verification is by GC/MS or FTIR analysis. Primary standards are utilized as reference standards in casework, research and development or methods, training, and quality control of critical reagents.

Secondary Standards are comprised of pharmaceutical preparations, plant materials (e.g. marijuana, peyote, commercial synthetic cannabinoid products, salvia, herbs, or mushrooms), some non-controlled drug standards, and selected retained casework samples. They normally do not enter the laboratory with a certification of purity, but are verified at some point by GC/MS or FTIR or are obtained from a reliable manufacturer/distributor or government agency. These standards are utilized for reagent QA (e.g. Weber Test positive control), research where purity is not crucial, training, or confirmation of a non-controlled substance.

Working Standards are dilute solutions of drugs prepared for instrumental use or small amounts (less than 500 mg) removed from inventory for QC on critical reagents. The removal of drug from audited inventory is recorded in the binder of drug standards at the time of initial preparation or subsampling, but after that these minute amounts of drug are not tracked for weight.

Training Materials are drug substances that are kept in locked storage but not used for any analytical purpose. Training drugs are primarily tablets and capsules, many of them clandestinely manufactured, taken from casework submissions. They are not inventoried and their function is for training and display.

A "Drug Standards" binder is kept in the controlled substances laboratory with drug standard information including:

- current inventory
- consumed drug standards (or record of destruction/expiration)
- QA verification

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The Drug Standards binder is used to document the verification of drug standards. The drug verification should include:

- Name of drug, approximate concentration, and diluent.
- Date prepared.
- Assigned lot number with initials of analyst preparing standard.
- Source of standard material with company lot number and/or product identification.
- Verification method (GC/MS or FTIR) and the initials of the Forensic Scientist performing verification.
- Expiration date (if applicable). Where a purchased chemical or reagent is already marked with an expiration date from the manufacturer, this date shall be effective unless an earlier date is chosen by the laboratory. A later date may also be chosen by the laboratory, but only if it can be demonstrated that there is no loss of performance at the time of usage. A preferable use of 'expired' drug standards is for qualitative training or demonstration exercises.

Drug standards are prepared as needed for instrumental analysis, usually to an approximate concentration of 1mg/mL in methanol. Since these are not used for quantitative analysis, an approximate concentration is sufficient. Each time a new standard is prepared or opened, it receives a lot number, utilizing either the manufacturer's lot number from solution ampules OR a lab-generated lot number, utilizing the convention "MMDDYY analyst initials".

Verification is by GC/MS or FTIR analysis. Primary standards are utilized as reference standards in casework, research and development or methods, training, and quality control of critical reagents.

Standards suspected of being contaminated or decomposed may no longer be used as a standard until re-authenticated.

Records: Drug Quality Certificates and any documents of authentication that are received with standards in hard copy form are kept in the Drug Standards binder.

Verification spectra of new drug standard lot numbers placed into service will be stored in the LIMS in the file CS INST 20XX.

The **Drug Standards** binder stores hard copy records of:

- Primary Standards inventory
- Non-Controlled and Secondary Standards inventory
- Consumed drugs
- Log of new **working standard** solutions prepared (with QA data) or small amounts (<500 mg) of drug removed for use in quality control of critical reagents.

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Removal of any quantity of primary or secondary drug standards from inventory will be recorded in the Drug Standards binder.

Drug inventory is also recorded electronically on the laboratory's computer network. This is updated yearly after the annual audit.

Security: The Scientific Crime Detection Laboratory is responsible for assuring that its drug standards are secure:

- All drug standards (controlled or uncontrolled) shall be stored in a locked and secure location.
- Access to drug standard locations shall be restricted to the controlled substances discipline supervisor and analysts, the Quality Assurance Manager, and the Laboratory Manager.
- An audit of all drugs will be made yearly, updates performed, and a memo written to the Laboratory Manager through the Quality Assurance Manager indicating that this audit was performed. This audit will account for all drugs within the laboratory and will include a yearly gross weight measurement of all controlled substances. The only exceptions to the audit mandate are Working Standards and Training Materials.

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Appendix I Abbreviations

ADA: Assistant District Attorney
AgNO₃: Silver Nitrate
APAP: acetaminophen (for *N*-acetyl-*para*-aminophenol)
APD: Anchorage Police Department
ATR: Attenuated Total Reflectance
AuCl₄: Gold Chloride
BATF & E: Bureau of Alcohol, Tobacco, Firearms, and Explosives
BB DCM: Borate Buffer / Dichloromethane
BD: 1,4-Butanediol
BZP: *N*-Benzylpiperazine
CBD: Cannabidiol
CBN: Cannabinol
CH₂Cl₂: Dichloromethane (**DCM** or Methylene Chloride)
2C-B: 4-Bromo-2,5-dimethoxyphenethylamine
2C-E: 4-Ethyl-2,5-dimethoxyphenethylamine
2C-I: 4-Iodo-2,5-dimethoxyphenethylamine
2C-T-7: 2,5-Dimethoxy-4-(*n*)-propylthiophenethylamine
Cu(NO₃)₂: Cupric Nitrate
DA: District Attorney
DAO: District Attorney's Office
DCM: Methylene Chloride (**CH₂Cl₂** or Dichloromethane)
DIB: Drug Identification Bible
DiP-5-MeOT: *N,N* Diisopropyl-5-methoxytryptamine
DiPT: *N,N*-Diisopropyltryptamine
DOC: 2,5-Dimethoxy-4-chloroamphetamine (or 4-Chloro-2,5dimethoxyamphetamine)
DOB: 2,5-Dimethoxy-4-bromoamphetamine (or 4-Bromo-2,5-dimethoxyamphetamine)
DOET: 2,5-Dimethoxy-4-ethylamphetamine
DOI: 2,5-Dimethoxy-4-iodoamphetamine
DOJ: Department of Justice
DPT: *N,N*-Dipropyltryptamine
d. WATER: distilled or deionized water
e-Case File: Electronic Case File
EtOH: Ethanol
FTIR: Fourier Transform Infrared Spectroscopy
FPD: Fairbanks Police Department
FBKS: Fairbanks
g: gram
GC/MS (or GC-MS): Gas Chromatography-Mass Spectrometry
GHB: Hydroxybutyric Acid
GBL: Gamma Butyrolactone
HAuCl₄: Gold chloride
HCl: Hydrochloric acid
HCO₃: Bicarbonate

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HI: Hydriodic Acid

H₂O: Water

4-HO-DIPT: 4-Hydroxy-N,N-diisopropyltryptamine

HP: Hewlett Packard

H₂SO₄: Sulfuric Acid

H₃PO₄: Phosphoric Acid

ICFI: Insufficient Concentration for Identification

IPA: Isopropyl Alcohol (or Isopropanol)

IR: Infrared Spectroscopy

ISFI: Insufficient Sample for Identification

ID: Identification

JWH-018: 1-naphthalenyl-(1-pentyl-3-indolyl)methanone

JWH-073: (1-butyl-3-indolyl)-(1-naphthalenyl)methanone

JWH-250: 2-(2-methoxyphenyl)-1-(1-pentyl-3-indolyl)ethanone

KBR: Potassium Bromide

kg: kilogram

lb(s): Pound(s)

Less than: <

LIMS: Laboratory Information Management System

LSD: Lysergic acid diethylamide

5-MeO-DIPT : 5-Methoxy-N,N-diisopropyltryptamine

5-MeO-DMT: 5-Methoxy-N,N-dimethyltryptamine

5-MeO-MIPT: 5-Methoxy-N-methyl-N-isopropyltryptamine

5-MeOT: 5-Methoxytryptamine

6-MeOT: 6-Methoxytryptamine

MeOH: Methanol

MDA: Methylenedioxyamphetamine

MDMA: Methylenedioxymethamphetamine

MDOH: N-Hydroxy-3,4-methylenedioxyamphetamine

mg: Milligram

mL: Milliliter

MMDA: 5-Methoxy-3,4-methylenedioxyamphetamine

MMDDYY or MMDDYYYY: 2 digit month/2 digit day/ 2 or 4 digit year

MSD: Mass Selective Detector

MSDS: Material Safety Data Sheet

MSM: Methylsulfonylmethane or Dimethyl Sulfone

MSTFA: N-methy-N-(trimethylsilyl) trifluoroacetamide (or N-trimethylsilyltrifluoroacetamide)

5-MT: 5-Methyltryptamine

7-MT: 7-Methyltryptamine

µl or µL: Microliter

N/A: Not Applicable

NA: Not Analyzed

NaOH: Sodium Hydroxide

Na₂SO₄: Sodium Sulfate

NCSD: No Controlled Substances Detected

NH₄OH: Ammonium Hydroxide

NFLIS: National Forensic Laboratory Information System

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NIST: National Institute of Standards and Technology
NSB PD: North Slope Borough Police Department
pck: Package
PD: Police Department
PDR: Physicians' Desk Reference
p-DAB: P-Dimethyl aminobenzaldehyde
Pet Ether: Petroleum Ether
PFTBA: Perfluorotributylamine
PMA: 4-Methoxyamphetamine
P-2-P: Phenyl-2-propanone
QA: *Quality Assurance*
QC: *Quality Control*
QIFA: Quantity Insufficient for Analysis
QNS: Quantity Not Sufficient
SOQ: Statement of Qualifications
STP: 2,5-Dimethoxy-4-methylamphetamine
TFMPP: Trifluoromethylphenylpiperazine
THC: Tetrahydrocannabinol
TLC: Thin layer chromatography
Triage: The practice or principle of allocating limited resources, prioritization
UAA: University of Alaska Anchorage
UAF: University of Alaska Fairbanks

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Appendix II

Weight Considerations in the Alaska Statutes

- MICS I 11.71.010
 - 1) delivers **any amount** of IA
 - 2) delivers **any amount** IIA or IIIA to person <19 years old

- MICS II 11.71.020
 - 1) manufactures or delivers **any amount** IA
 - 2) manufactures any **amount** of methamphetamine
 - 3) possesses more than **6 grams** of ephedrine or pseudoephedrine with intent to manufacture

- MICS III 11.71.030
 - 1) manufactures or delivers **any amount** IIA, IIIA
 - 2) delivers **any amount** of IVA, VA, or VIA to a person less than 19 years old

- MICS IV 11.71.040
 - 1) manufactures/delivers **any amount** of IV or VA
 - 2) manufactures or delivers a weight of **1 ounce** or greater VIA
 - 3) possesses **4 ounces** or greater of VIA
 - 4) possesses **500 mg or more** of IIA (11-15), substituted cathinones (bath salts).

- MICS V 11.71.050
 - 1) manufactures or distributes **less than 1 ounce** VIA
 - 2) possesses **greater than 3 grams** IIIA or IVA
 - 3) possesses **greater than 1 ounce** VIA

- MICS VI 11.71.060

possesses **less than 1 ounce** VIA

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Appendix III Guidelines for Technical and Administrative Reviews

TECHNICAL REVIEW	Analyst:	Date:
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Yes N/A

- The correct laboratory number appears on each page of the case file.
- The analyst's initials or name appear on all pages of the case file.
- Sub-itemizations are clearly labeled and consistent throughout LIMS, bench notes, and report.
- For each controlled substance identified, a standard was analyzed and is included in the notes.
- For each GC/MS or FTIR evidence spectra, a blank is included in the notes.
- Evidence retention times and spectra agree with standard retention times and spectra.
- 'Physical identifications only' have adequate documentation.
- All reference or internet searches are documented in the case file.
- The worksheet indicates the dates that analysis was started and completed.
- Each item's packaging is documented.
- The officer's description of items agrees with the analyst's description.
- The weights or counts on the worksheet and the report are in agreement and the weights are truncated in the report.
- The type of analysis and results are documented for each item in the bench notes.
- All chemical testing and all physical identification searches are documented in the notes.
- Two samplings are used for all conclusive identifications.
- An appropriate extraction/clean-up procedure was employed.
- The analysts' conclusions in the report are supported by documentation in the notes.
- The item descriptions agree between LIMS, bench notes, spectra, and report.
- The electronic chain of custody in LIMS agrees with the worksheet and report.

ADMINISTRATIVE REVIEW	Analyst:	Date:
-----------------------	----------	-------

Yes N/A

- The requesting agency, agency file number, and lab number agree with the lab submittal.
- The correct name of the submitting officer and/or whom to reply to appears on the report.
- The lab item number and description of the item are correct.
- Items examined but not analyzed are included in the report Description & Results.
- Grammar, spelling, and punctuation are correct.
- The report is signed by the analyst.
- Any latent print request is changed from "Latents Pending" to "Latents Processing" and re-dated.
- Report is referred appropriately for distribution to agency.

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Appendix IV Hypergeometric Sampling Plan

# of UNITS	SCREEN & CONFIRM
1-10	All
11-12	10
13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-939	28
940+	29

Hypergeometric Sampling

- Based on statistical probability, there is a 95% likelihood that at least 90% of the units contain the drug.
- Visual similarity adds another level of confidence. If a sampling plan is applied, the expert's opinion is that all units are the same substance.
- After confirming 29 units, there is no significant increase in confidence level.
29 units = ~6 hours of instrument time
- If any results are different than the rest, The analyst must re-evaluate the population.

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Appendix V Uncertainty of Measurement

(Policies to be determined)

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Appendix VI SWGDRUG Recommendations

See I:\Discipline Shares\Controlled Substances\SWGDRUG Guidelines

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Appendix VII Revision History

This Drug Chemistry Procedure Manual represents a major re-write of the Controlled Substances Procedure Manual with input from the Forensic Laboratory Manager. All changes are not included in the revision history but major differences are addressed:

Section(s) Revised	Date	Approving Authority
Section 1.3, Sample Conservation , p. 5, changed "Insufficient Concentration for Identification" to "Insufficient quantity for complete identification" will be reported.	6-25-2012	Jane Booth, Supervisor
Section 1.4, Identification Requirements , page 5, changed "NOTE: Identification of a non-controlled drug (e.g. acetaminophen, ibuprofen, guaifenesin, or diphenhydramine) in a pharmaceutical preparation is exempt from the requirement that a standard be analyzed concurrently. When identification may be important in determining the charging statute, a library search of previous GC/MS spectra along with a literature search of the pharmaceutical is sufficient" to: "A commercial preparation may be identified by reference material only (ex: by manufacturer logo), but shall clearly indicate in the report that identification was by "physical identification only, no chemical testing was performed."	6-25-2012	Jane Booth, Supervisor
Section 1.4, Identification Requirements , p. 5, deleted "Reporting "No Controlled Substances per Alaska Statutes" may be accomplished by means of a single structurally-elucidating analysis (utilizing an appropriate screening program) OR by reference literature findings which allow identification of the drug as one that is uncontrolled in Alaska." Added "For negative samples, the testing process between samplings shall be different enough to minimize the possibility of a false negative. Example, a sample may be analyzed by GC/MS with a simple dilution in solvent, and a second sample extracted from buffer to solvent, to ensure a salt, acid, or basic form of a substance is not missed."	6-25-2012	Jane Booth, Supervisor

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Section 2.4, Color Tests , p. 10, changed every 6 months to every year. Added guidelines for use of NIK test kits.	6-25-2012	Jane Booth, Supervisor
Section 2.5, Crystal Tests , p. 13, deleted gold chloride/phosphoric acid test for methamphetamine.	6-27-2012	Jane Booth, Supervisor
Section 3.1, GC/MS, Interpretation of Data , p. 29, Added '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.'	6-27-2012	Jane Booth, Supervisor
Section 3.1, GC/MS, Interpretation of Data , p. 29, deleted 'Comparison of retention time data between evidence standards and primary standards must demonstrate agreement within $\pm 5\%$ or 0.05 minutes (3 seconds).'	6-27-2012	Jane Booth, Supervisor
Section 3.1, GC/MS, U of M and Good Laboratory Practice , p. 29, deleted 'Placement of autosampler vials is checked before starting sequence.'	6-27-2012	Jane Booth, Supervisor
Section 3.2, FTIR, Quality Control , p. 34, deleted "periodically and" from 'Instrument validations are run after any major service or hardware replacement.	6-28-2012	Jane Booth, Supervisor
Section 3.2, FTIR, Procedures , p. 34, deleted procedures for KBr sample preparation.	6-28-2012	Jane Booth, Supervisor
Section 3.3, Balances, Quality Control , p. 34, updated balance information.	6-29-2012	Jane Booth, Supervisor
Section 4.3, FTIR, Routine Disclosure , p. 40, deleted .	6-28-2012	Jane Booth, Supervisor
Appendix, Weight Considerations in the Alaska Statutes , p. 48, added substituted cathinone legislation.	6-29-2012	Jane Booth, Supervisor