



Ada County Sheriff's Office

Forensic Lab

Drug Chemistry Analytical Method

Version 2.0

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1.0 Scope

- 1.1. Controlled substances are commonly found in solid samples, liquid samples and plant material, and vary in size and concentration.
- 1.2. Established performance based methods (guidelines) for the analysis of submitted substances, have been developed because of this diversity.
- 1.3. These procedures allow the individual scientist the flexibility to choose the best methods with which to analyze substances submitted.
- 1.4. ACSO Forensic lab will perform quantitative analysis on methamphetamine only.

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2.0 References

- 2.1. ACSO Policy Manual located at <https://aces.ada.net.gov/sites/ADAPortal/departments/sheriff/policy/Documents/ACSO%20Policy%20Manual%20112414.pdf>
- 2.2. Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 2.3. Ada County Sheriff's Office Forensic Lab Health and Safety Manual
- 2.4. SWGDRUG located at <http://www.swgdrug.org/>
- 2.5. NIST OSAC Subcommittee: Seized Drugs at <http://www.nist.gov/forensics/osac/sub-drugs.cfm>
- 2.6. College/University level Chemistry text books
- 2.7. Operation and maintenance manuals for each analytical instrument

3.0 Terms and Definitions

- 3.1. **Accuracy** -the extent to which a given measurement agrees with the standard value for that measurement.
- 3.2. **Aliquot** – A sample or portion of a total amount.
- 3.3. **Anabolic steroid** – Synthetic substances related to the male sex hormones that promote the growth of skeletal muscle and development of male sexual characteristics (anabolic-androgenic steroids).
- 3.4. **Analytical balance** – A class of balance designed to measure small mass in the sub-milligram range.
- 3.5. **Background spectrum** – Measures the response of the spectrometer without a sample in place, and is subtracted out.
- 3.6. **Balance** – A weighing scale used to measure weight or calculate mass.
- 3.7. **Base peak** – most abundant ion formed in the ionization chamber.
- 3.8. **Binomial distribution** – A discrete probability distribution used to model the number of successes in a sample of size n drawn with replacement from a population of size N .
- 3.9. **Certified weight** – precision weight with documentation that it meets a certain standard.
- 3.10. **Clandestine** – secret and concealed, often for illicit reasons.
- 3.11. **Composite** – combining of visually similar evidence items into a single tem for analysis.
- 3.12. **Coning and quartering** – method used to reduce the sample size without creating a systematic bias, involves pouring sample so it takes on a conical shape then dividing it into quarters, 2 opposite quarters are removed while the other 2 opposite quarters are combined, these are further reduced down by the same method until an appropriate sample size remains.
- 3.13. **Controlled substance** – A listed compound found either in Idaho Statutes Title 37 Chapter 27 or the Code of Federal Regulations Title 21 Part 1308.
- 3.14. **Control sample** - A comparison standard for verifying or checking the findings of an experiment. A positive control contains the analyte of choice for the method or test being evaluated. A positive control is used to evaluate a positive result. A negative control does not contain the analyte of choice and is used to verify a negative result.

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- 3.15. Depressant** – A substance which depresses the central nervous system. Effects include sedation, muscle relaxation, pleasure, removal of anxiety, and an overall sense of calmness.
- 3.16. Derivatization** – a technique used in chemistry which transforms a chemical compound into a product (the reaction's derivative) of similar chemical structure, called a derivative
- 3.17. Diastereomer** – one of two stereoisomers that are not mirror images of each other and are non-superimposable on one another
- 3.18. Distribution** – process of making a product available for use or consumption
- 3.19. Drug** – medicine or other substance which has a physiological effect when taken.
- 3.20. Enantiomer** – one of two stereoisomers that are mirror images of each other that are non-superimposable on one another
- 3.21. Expanded uncertainty** – multiplying the combined standard uncertainty by a coverage factor, k , to give a level of confidence.
- 3.22. Gain** – How much the detector signal is amplified electronically. It affects the ratio of DC voltage to RF frequency on the mass filter which controls the width of the mass peaks. A higher gain yields narrower peaks (more so at higher masses).
- 3.23. Hallucinogen** – A class of substances which affects the senses and perception of an individual. Sights and sounds are changed or enhanced, sense of time is distorted.
- 3.24. Hash/Hashish** – Collection of the oily, THC-rich resin glands found on all exterior parts of the marijuana plant, except the seeds.
- 3.25. Hash Oil** – A concentration of the oil contained in the resin glands of the marijuana plant.
- 3.26. Heterogeneous** – Non-uniform in composition or character.
- 3.27. Homogeneous** – Uniform in composition or character.
- 3.28. Hypergeometric distribution** – A discrete probability distribution that describes the probability of k successes in n draws, *without* replacement, from a finite population of size N that contains exactly K successes, wherein each draw is either a success or a failure.
- 3.29. Ion source** – one of the major components of a Mass Selective Detector (MSD) analyzer, device that creates atomic and molecular ions, either by electron impact (EI) or chemical ionization (CI).

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- 3.30. Instrument blank** - A volume of clean solvent that is analyzed on an instrument to ensure that the instrument is working properly and/or that there is no contamination issues associated with the instrument.
- 3.31. Interferogram** – graph on the FTIR which shows constructive/destructive interference.
- 3.32. Internal standard** – substance added (in a constant amount) to all samples in an analysis to help compensate for several types of random and systematic errors and to obtain an analyte concentration in a calibration curve.
- 3.33. Isomer** – molecules that have the same molecular formula but have a different arrangement of the atoms in space.
- 3.34. Isotope** – atoms of a given element which differ from each other in the number of neutrons in the nucleus, their electronic structure being the same.
- 3.35. Manufacture** – production, preparation, propagation, compounding, conversion or processing of a controlled substance.
- 3.36. Method blank** - An analytical control consisting of all reagents and solvents that is carried through the entire analytical procedure.
- 3.37. Molecular ion** - In the mass spectrum, the heaviest ion (the one with the greatest m/z value)
- 3.38. Narcotic** – Medicinally, a compound which dulls the senses, relieves pain, and produces a slumber. Has been used to refer to all controlled substances at times.
- 3.39. Narcotic drug** – Drug made from opiates and opioids.
- 3.40. Normal distribution** – A bell curve showing values are more likely to fall near the average than further away (Gaussian).
- 3.41. Opiate** – A naturally occurring compound isolated from raw opium or a semi-synthetic compound produced from a product contained in raw opium.
- 3.42. Opioid** – A fully synthetic compound which has central nervous system properties similar to an opiate.
- 3.43. Population** - The collection of items under discussion. A population may be real or hypothetical; finite or infinite; homogeneous or heterogeneous.
- 3.44. Primary standard** - A commercially purchased compound that is traceable back to a manufacturer. Its identity is confirmed by verifying its composition through a comparison of FTIR or GCMS with literature, a certificate of analysis or a previously confirmed primary standard.

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- 3.45. Precision** - the extent to which a given set of measurements of the same sample agree with their mean.
- 3.46. Racemic** - mixture that is composed of equal amounts of dextrorotatory and levorotatory forms of the same compound
- 3.47. Random sample** - A sample chosen from a population without bias, each sample is chosen randomly and entirely by chance, such that each sample has the same probability of being chosen at any stage during the sampling process.
- 3.48. Reagent** – a substance used because of its chemical or biological activity or because it takes part in or brings about a particular chemical or biological reaction.
- 3.49. Reference material** – Material or substance, one or more of whose property values are sufficiently homogenous and well-established to be fit for its intended use in measurement or in examination of nominal properties (i.e. drug standards).
- 3.50. Relative abundance** – percentage of abundance of other peaks in relation to abundance of the base peak.
- 3.51. Representative sample** – A sample of a statistical population that accurately reflects the members of the entire population.
- 3.52. Residue** - samples which are either too small to be weighed accurately or that which remains after the bulk has been removed.
- 3.53. Resolution** – how close two peaks can be and still be identified as separate peaks.
- 3.54. Root sum squares** – the square root of the sum of the squares of a series of related values
- 3.55. Salt** – ionic compound composed of positive ions and negative ions, a crystalline material
- 3.56. Sample** - A unit or a number of units selected from a population. Taking a part of a substance, material or product for testing in order to reach a conclusion, make an inference about, and report on the whole. Sampling should only be used when there is a reasonable assumption of homogeneity of the whole.
- 3.57. Sampling plan** – For an item that consists of a multi-unit population (tablets, baggies, bindles), a sampling plan is a statistically valid approach to determine the number of sub-items that must be tested in order to make an inference about the whole population.
- 3.58. Sampling procedure** – A defined procedure used to collect a sample or samples from the larger whole, to ensure that the value obtained in the analysis is representative of the whole. The sampling procedure may include details about size and number of

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samples(s) to be collected, locations from which to collect the samples(s), and a method to ensure the homogeneity of the larger whole (or to make it so).

- 3.59. Sampling selection** – A practice of selecting items to test, or portions of items to test, based on training, experience and competence. In sample selection, there is no assumption about homogeneity.
- 3.60. Secondary standard** - A laboratory produced sample or case work sample whose composition has been verified by comparison of its Category A instrumental data (e.g., GC/MS, FTIR, or Raman) to that of a primary standard or a scientifically accepted literature reference.
- 3.61. Smoothing** – used to improve the appearance of the spectrum obscured by noise.
- 3.62. Solvent blank** - A negative control of any solvent that could potentially be used in an analytical scheme.
- 3.63. Standard uncertainty** – a margin whose size can be thought of as “plus or minus one standard deviation”, tells us about the uncertainty of an average.
- 3.64. Stereoisomers** – molecules that have the same molecular formula and sequence of bonded atoms but differ in the three-dimensional orientations of their atoms in space.
- 3.65. Stimulant** – A substance which stimulates the central nervous system. Effects include an increase in energy, pleasure, satisfactions, or alertness and possibly a decrease in appetite.
- 3.66. Tare** – Reset the zero of the scale display when an empty container is placed on the weighing platform, in order to display only the weight of the contents of the container.
- 3.67. Trace amount** – refer to Residue.
- 3.68. Truncate** – to shorten (a number) by dropping one or more digits after the decimal point
- 3.69. Uncertainty of measurement** – an estimated value, within a specified confidence limit, that depicts a value of variability that can be attributed to the result or test.
- 3.70. Unit** - One member of a population made up of visually similar evidence items. For example, 1000 bindles are submitted and comprise the population. A unit would be one bindle.

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4.0 Management Requirements

- 4.1. **Organization** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.2. **Management system** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.3. **Document control** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.4. **Review of requests, tenders, contracts** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.5. **Subcontracting tests** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.6. **Purchasing Services and Supplies**
 - 4.6.1. Purchasing documents shall indicate that the defined specifications are met for the critical supplies listed below.
 - 4.6.1.1. Ultrapure Helium gas.
 - 4.6.1.2. Drug standards.
 - 4.6.1.3. Spectroscopic grade solvents or better
 - 4.6.1.3.1. Chloroform stabilized with Ethanol or Pentene.
 - 4.6.1.4. Balance calibrations
- 4.7. **Service to customer** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.8. **Complaints** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.9. **Nonconforming work** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.10. **Improvement** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.11. **Corrective action** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual
- 4.12. **Preventive action** See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

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4.13. Control of records

- 4.13.1.** The documentation needed to support the conclusion(s) in the report shall be kept in the case file. Current batch documentation shall be stored in an area of the laboratory known to and accessible to the drug chemists.
- 4.13.2.** Authentication documentation shall be kept for each standard.
- 4.13.3.** GCMS and FTIR instrumental data files shall be backed up monthly.
- 4.13.4.** A maintenance log shall be kept for the GCMS and FTIR. Entries into the log should include any symptoms and type of maintenance performed.
- 4.13.5.** The approved abbreviation list for the drug chemistry discipline can be found in the electronic lab documents. Commonly accepted abbreviations or abbreviations that are readily recognizable to a reviewer do not need to be listed, such as those for metrics or elements. Chemical formulas do not need to be listed as abbreviations, but shorthand notations such as MeOH, for methanol, do. Care should be taken when isomers may be involved. For the designer drugs, a chemist should refer to professional literature for the accepted abbreviation.
 - 4.13.5.1.** Should an analyst use an abbreviation that is not listed in the abbreviation list, then a reference shall be included in the case notes as to what the abbreviation means.

4.14. Internal audits See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

4.15. Management reviews See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

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5.0 Technical Requirements

5.1. General

5.1.1. A positive and negative control shall be run to verify the reagent preparation. This control shall occur before use or, if appropriate, concurrent with the test.

5.1.1.1. The positive control shall be performed by testing the reagent with a substance that will react with the reagent to produce a known color.

5.1.1.2. A negative control shall be performed by testing the reagent with a substance known not to react or with a blank spot well.

5.1.1.3. The results of these tests shall be recorded, along with the date the reagent was made, the initials of the preparer, and if the reagent worked as expected. This shall be recorded on the reagent log.

5.1.1.4. All color reagents that are made in bulk shall be checked monthly and the results of the checks shall be recorded. EXCEPTION: Duquenois-Levine or other color reagent made in bulk that is checked with each use.

5.1.1.4.1. Bulk reagents include; Marquis, Simons, and Cobalt Thiocyanate.

5.1.1.4.2. A methamphetamine standard shall be used for the positive control for the Marquis and Simons color reagent. A cocaine standard shall be used for the positive control for the Cobalt Thiocyanate color reagent.

5.1.1.4.3. Dimethyl sulfone shall be used for the negative controls.

5.1.1.5. The control results for reagents that are prepared for one time use shall be documented in the case notes.

5.1.1.6. In the case notes, a "positive control" indicates both the controls for the standard and the blank worked properly. A "negative control" indicates one or both controls were unsuccessful.

5.1.1.7. Reagents prepared in the laboratory shall be labeled with the identity of the reagent, date of preparation and/or lot number, and identify of who made the reagent.

5.1.1.7.1. For a prepared reagent, the lot number shall be the date and the initials of the analyst who prepared it (i.e. 070716HC).

5.1.1.8. The components used in the preparation of the reagent shall be documented on the reagent log, as well as who made the reagent.

5.2. Personnel See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

5.3. Accommodation and Environmental Conditions

5.3.1. Standard laboratory safety protocols should be followed. Refer to the ACSO Forensic Lab Health and Safety Manual.

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- 5.3.2.** To prevent contamination of personnel and the exhibit, the appropriate equipment should be utilized (e.g., gloves, eye protection, etc.) To minimize cross-contamination, personal protective equipment should be worn and changed when necessary.
- 5.3.3.** Appropriate eye protection shall be worn whenever a hazard to the eyes exists. This would include chemical exposures and alternate light sources, (UV, laser, etc.).
- 5.3.4.** Examination utensils (e.g., spatulas, forceps, scissors, measuring devices, etc.) used in processing should be cleaned between items of evidence.
- 5.3.5.** When using aerosol type chemicals, proper engineering controls, work practice controls, or PPE must be employed to avoid chemical exposure.

5.4. Testing Methods and Method Validation

5.4.1. Physical Examination

- 5.4.1.1.** After removing the sample from its packaging (if appropriate), a thorough physical characterization of an exhibit should be conducted and the following observations should be noted if applicable: type of material, color, size, shape, amount, morphology, significant markings, odor and texture.

- 5.4.1.1.1.** Pills/tablets shall have the entire imprint/logo documented.
Documentation can be written, sketched, photographed or photocopied.

5.4.2. Weighing

- 5.4.2.1.** At some point during the physical examination of the item, a weight or count must be determined. Prior to weighing a sample, the analyst shall make sure the balance is clean and tared.

- 5.4.2.1.1.** Exceptions to this include when dealing with residue amounts or a sample whose nature precludes an accurate weighing (sticky, sludge, moldy, etc.).

- 5.4.2.1.2.** The reason why it was not weighed must be in the case notes.

- 5.4.2.1.3.** Residue or trace shall be defined as anything less than 0.10 grams.

- 5.4.2.2.** Items that contain a small amount of material that clings to the packaging can be considered residue and reported as such.

- 5.4.2.2.1.** Smoking devices that contain a substance packed into the bowl (regardless of the amount) do not have to be removed or weighed and can be reported as residue.

- 5.4.2.3.** Net weights are determined by weighing only the contents of a container.

- 5.4.2.4.** Gross weight measurements are made by weighing the entire item (packaging and contents).

- 5.4.2.5.** All digits observed from the balance shall be recorded in the case notes.
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5.4.2.6. When the **total** weight of a sample(s) falls within the window of uncertainty at regulatory limits, then the uncertainty associated with each weighing event must be listed on the report. **See Section 5.6.6.**

5.4.3. Categorizing Analytical Techniques

5.4.3.1. Techniques for the analysis of drug samples are classified into three categories (see Table 1) based on their maximum potential discriminating power. However, the classification of a technique may be lower, if the sample, compound, or mode of operation diminishes its discriminating power. An example of diminished discriminating power is an infrared spectroscopy technique applied to a mixture which produces a combined spectrum.

Table 1: Categories of Analytical Techniques

Category A	Category B	Category C
Fourier Transform Infrared Spectroscopy (FTIR)	Gas Chromatography (GC)	Color Tests
GCMS	Mass Spectrometry (MS)	
	Thin Layer Chromatography (TLC)	
	Pharmaceutical Identifiers	
	Cannabis only: Macroscopic Examination Microscopic Examination	

5.4.4. Identification Criteria

5.4.4.1. When a Category A technique is incorporated into an analytical scheme, then at least one other technique (from either Category A, B, or C) shall be used, if possible.

5.4.4.2. When a Category A technique is not used, at least three different validated techniques shall be employed.

5.4.4.3. This combination shall identify the specific drug present.

5.4.4.4. For quality assurance purposes, when sample size allows for it, the second technique should be applied on a separate sampling. Case notes shall specify when multiple techniques are applied to the same sampling and why.

5.4.4.5. If only one sampling event can be performed on a sample, then an n-tridecane internal standard (IS) shall be added to the extract before analysis on the GCMS. A blank with internal standard shall be run prior to the extract.

5.4.4.5.1. Add a couple drops of the IS (1.3mL of n-tridecane to 1L of chloroform) to the sample and blank vials prior to analysis.

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- 5.4.4.5.2.** Caution, the IS is volatile. Care must be taken when analyzing the samples to ensure there is an adequate amount of the IS for detection.
- 5.4.4.6.** In order to report "No Controlled Substance(s) detected", at a minimum, a sample must be run on the GCMS using a temperature program and extraction scheme that encompasses a wide range of drugs.
- 5.4.4.7.** A sample shall be defined as any casework related extract, solution, or solid that is not returned to evidence.
- 5.4.4.8.** If a compound is derivatized as part of the analytical scheme, the GCMS of the non-derivatized sample in combination with the GCMS for the derivatized sample is considered two separate tests in Category A, provided that they are from two separate samplings as described above.
- 5.4.4.9.** Before a drug standard can be used as a reference for casework, it shall be authenticated, either on the GCMS or FTIR.
- 5.4.4.9.1.** A standard shall be considered authenticated when the library match is greater than 85%. If the match is less than 85%, then two chemists must concur on the validity of the match and initial the printout. This printout shall be kept in the standards logbook or saved electronically.
- 5.4.4.9.2.** Reference libraries can come from any reliable source, including, but not limited to, instrument libraries, scientific journals or publications. When a reference is not available, mass spectral interpretation may be used in conjunction with the Certificate of Analysis (COA). This would apply in cases where instrumental data for a drug metabolite or isomer is not yet published, but a structurally similar compound is available to assist with interpretation. Again, this would require two chemists to concur on the interpretation and initial the printout and the printout shall be kept in the standards logbook or saved electronically.
- 5.4.4.10.** Confirmatory data from Category A techniques (Table 1) shall be compared to an authenticated drug standard ran within 24 hours of running the unknown.
- 5.4.4.11.** All analytical techniques shall have documentation that is reviewable. Examples of reviewable documentation include:
- 5.4.4.11.1.** Spectra, chromatograms, and photographs/photocopies of TLC plates.
- 5.4.4.11.2.** Pharmaceutical identifiers (i.e. notes shall include Drugs.com, Drug ID Bible or other identifier page(s)).
- 5.4.4.12.** For any method to be considered of value, the test must be considered "positive". While "negative" tests provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the forensic identification of a drug.

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5.4.4.13. If an analyst has met or exceeded the requirements for the identification of a substance, the report shall simply state the result, and the Schedule (if applicable) in a section easily identifiable as "Findings", "Conclusions", or similar wording.

5.4.4.14. For investigative purposes, a sample can be hand delivered to the laboratory and analyzed with color tests and/or on the GCMS or FTIR. If instrumentation is utilized, the result shall be compared to a library search. This analysis will be documented in case notes.

5.4.4.14.1. If confirmation is required, an aliquot may be retained to perform further testing

5.4.4.14.2. A report shall be issued for any analysis performed in the laboratory. If a confirmation test is not run, the report shall clearly state that the results are preliminary.

5.4.5. Minimum Examination Requirements

5.4.5.1. All controlled substances charged as felonies, in the State of Idaho, should be confirmed, if possible. **Exceptions are:** inadequate sample size, inability to obtain a standard, and compounds that are of the same drug class that have an instrumental response that is relatively minor compared to the major peak and/or can reasonably be assumed to be a byproduct of the manufacturing process. Examples include, but are not limited to, morphine and codeine in a heroin sample or P2P from a suspected clan lab.

5.4.5.2. If a case has a mixture of compounds from different schedules, it is up to the analyst to determine what warrants confirmation. If it does not, then the report shall note that the sample indicates the presence of another controlled substance that was not confirmed.

5.4.5.3. All controlled substances should be scheduled, if possible. **Exception;** liquid sample in unmarked bottle containing a controlled substance, where the schedule is dependent on the concentration of that controlled substance. If liquid sample comes in a labeled pharmacy bottle and the results of analytical testing confirm the presence of the ingredients on the label, then the schedule on the label shall be reported.

5.4.5.4. Reporting of non-controlled substances shall be left up to the discretion of the analyst.

5.4.5.5. If control samples (solvent blanks) are submitted with syringe washes, they shall be treated as regular samples.

5.4.6. General Information

5.4.6.1. Attempts should be made to preserve evidence. The analyst shall make an effort to ensure enough of the evidence remains after analysis to allow for re-examination.

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- 5.4.6.2.** When only a trace level of sample is present, every effort shall be made to use less than one half of the sample. If it is necessary to use the entire sample, then any extracts, left over liquids, or residues shall be returned to the evidence envelope.
- 5.4.6.3.** Any extracts (washes) from evidence that did not have any visible residue shall be returned with the evidence.
- 5.4.6.4.** Laboratory notes shall contain the following, if applicable:
- 5.4.6.4.1.** A description of the packaging (plastic bag, glass vial, bindle, etc.).
 - 5.4.6.4.2.** A description of the evidence (powder, liquid, plant material, tablet including color, shape and imprint).
 - 5.4.6.4.3.** Weight or count (tablets/capsules) of evidence.
 - 5.4.6.4.4.** Balance used for weighing.
 - 5.4.6.4.5.** Reserve weight or count.
- 5.4.6.5.** GCMS spectra shall include:
- 5.4.6.5.1.** Instrument name
 - 5.4.6.5.2.** Date of analysis.
 - 5.4.6.5.3.** Method utilized.
 - 5.4.6.5.4.** Case number, exhibit number and analyst's initials or Manufacturer and/or lot number of the drug standard.
- 5.4.6.6.** FTIR spectra shall include:
- 5.4.6.6.1.** Instrument name.
 - 5.4.6.6.2.** Date of analysis.
 - 5.4.6.6.3.** Operating parameters.
 - 5.4.6.6.4.** Case number, exhibit number and analyst's initials.
- 5.4.6.7.** In multi item samples, the report shall clearly state what, and how many, items were tested. Example: "Three white oval tablets with imprint "M367", analyzed one."
- 5.4.6.8.** If an analytical scheme is employed that is restricted to a limited range of compounds, that limitation shall be clearly stated on the report along with any qualifiers. Example: "No basic drugs detected, examples of basic drugs include opiates, amphetamines, and cocaine."

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5.4.7. Sample Selection Rules

- 5.4.7.1.** Sample selection rules allow for the analysis of key evidence items within a case to maximize the resources of the lab. Requests for analysis on items not originally tested shall be reviewed and prioritized on a case by case basis.
- 5.4.7.2.** A felony charge has priority over a misdemeanor. Example: a gram of cocaine found in a suspect's pocket shall be tested while a gram of marijuana found in the same pocket may not be.
- 5.4.7.3.** A misdemeanor is treated equally to a felony if it is closer to the suspect or was the probable cause for a subsequent search. Example: A gram of marijuana found in a suspect's pocket would be analyzed in addition to a gram of cocaine found in the suspect's car.
- 5.4.7.4.** Based on the analysts training and experience, if it is suspected that different types of felony drugs are submitted then one of each type shall be analyzed. The analyst may use resources such as: statements of fact, description of items as well as visual inspection of items in making this determination.
- 5.4.7.5.** The analyst shall always strive to provide evidence supporting the highest charge, i.e. trafficking, manufacturing, delivery vs. felony possession vs. misdemeanor possession.
- 5.4.7.6.** For less than trafficking amounts, the number of samples necessary to support the charge shall be analyzed. Example: If you have five samples and the charge is possession, then only one sample needs to be tested. If the charge is intent to deliver, then more samples may need to be tested. Consultation with the prosecutor and/or officer should determine the number needed. The report shall state the total number of samples, the sample weight of the number actually analyzed, and the findings.
- 5.4.7.7.** For trafficking amounts, **ALL** samples shall be analyzed until the appropriate trafficking level is reached. Example: Forty balloons are submitted, each with about 0.1g of suspected heroin, the analyst shall continue to test balloons until they reach the first trafficking level, 2.0g.

5.4.8. Pharmaceuticals

- 5.4.8.1.** Pills that have recognizable logos and/or identification numbers need analytical confirmation if a literature search indicates that they contain a Schedule II controlled substance. **Exception**, if a controlled substance has been analytically confirmed from a non-pill sample in the case, then a pill(s) listed to contain the same controlled substance only needs a literature search. If a literature search reveals that pills with two, or more, different labels contain the same controlled substance, than only one of the pills needs to be analyzed. For the purpose of satisfying the identification criteria, the search shall be considered a presumptive test.
- 5.4.8.1.1.** While pharmaceutical identifiers are typically a reliable indicator of the content of a tablet, there are known instances of counterfeit tablets and

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errors in reference material. Analysts should take care to observe and note any abnormalities in color, size or markings that may reveal that the item is not a legitimate product. If a counterfeit is suspected or discovered, the evidence is no longer treated as a pharmaceutical in nature.

5.4.8.2. Acceptable literature references are published books (PDR, DIB, Logo index etc.), manufacturer's websites, Drugs.com, and labels from pharmaceutical packaging.

5.4.8.3. All literature searches shall have the source documented in the case notes.

5.4.8.4. Information from Poison control centers and non-manufacturers' websites can be used as a preliminary test when further analytical testing is performed or in conjunction with published books or approved websites to delineate pills with similar imprints and descriptions.

5.4.8.5. Sealed pharmaceutical packaging (i.e. manufacture-sealed boxes, blister packs, and foil pouches) may be used as a presumptive pharmaceutical identifier if there is no indication of tampering.

5.4.8.6. When analyzing a pharmaceutical tablet where some portion of the tablet or manufacturer's logo is missing, care should be taken when determining whether the remaining logo portion is sufficient to use as a Category B test (Table 1). Generally, a partial logo should not be used as a presumptive test. However, a logo may be sufficiently unique that the remaining logo portion is still readily identifiable. In those cases, the partial logo may be used as a presumptive identifier. Analysts should record other physical characteristics of the tablet that could assist in its identification (e.g. color, shape, dimensions, etc.).

5.4.8.7. A sample from each type of two-part, unsealed, gelatin type capsules shall be analyzed.

5.4.9. Color Tests

Chemical color tests produce characteristic color reactions that provide information regarding the nature of the substance being tested. Certain compounds or classes of compounds produce distinct colors when brought into contact with various chemical reagents. These simple reactions can indicate the presence of a particular functional group or molecular moiety. Colors are subjective and can be influenced by the concentration of the sample, the presence of contaminants, or by the age of the reagent.

5.4.9.1. Supplies (equipment/reagents/standards)

5.4.9.1.1. Spot plates, test tubes, spatulas.

5.4.9.1.2. Color test reagents, H₂SO₄, chloroform (commonly stored in dropper bottles), UV light.

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5.4.9.2. Reagent preparation

5.4.9.2.1. Directions for preparation of the commonly used color test reagents and the results can be located in Clarke's Analysis of Drugs and Poisons or other appropriate sources. The reagent log shall document the recipe used.

5.4.9.3. Method

5.4.9.3.1. Place 1-2 drops of reagent in a depression of a spot plate.

5.4.9.3.2. Add a small amount of sample.

5.4.9.3.3. Observe and record results.

5.4.9.3.4. Some tests require the use of a test tube instead of a spot plate (modified Duquenois-Levine or Scotts test).

5.4.9.4. Interpretations

5.4.9.4.1. Color test results shall be recorded in the case notes by detailing the color or lack of color reaction that results. If other reactions besides color are observed, they should also be recorded (i.e. effervescence, fuming, precipitation).

5.4.9.5. Cautions

5.4.9.5.1. If there is reason to suspect that the reagent is old or no longer working properly, it should be tested with a substance which will react with the reagent to produce a color. If the reagent does not produce the expected color with this substance, the reagent shall be discarded.

5.4.9.6. Controls

5.4.9.6.1. A blank (negative control) shall be run before each test. Place the appropriate reagent in a blank well and observe reaction (or lack thereof) prior to placing sample into well. Ensure you have a blank test prior to proceeding with addition of the sample. Refer to 5.1.1.2.

5.4.9.6.2. A positive control shall be tested when a reagent is made (and monthly, if spelled out in 5.1.1.4.1).

5.4.9.6.3. The results of one time use controls shall be documented in the case notes.

5.4.10. Thin Layer Chromatography (TLC)

5.4.10.1. Supplies (equipment/reagents/standards)

5.4.10.1.1. Thin layer chromatography tank and silica gel plates.

5.4.10.1.2. Aerosol spray units, capillary tubes, test tubes.

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5.4.10.1.3. Developing sprays, solvents (many different types may be employed depending upon the compound of interest).

5.4.10.2. Methods

5.4.10.2.1. A pencil-drawn line is placed on the TLC plate approximately 1-2 cm from the bottom of the plate, creating an origin line. The sample(s) and standard(s) are spotted on this line, allowing adequate space between the sides of the plate and between the samples themselves such that the samples do not migrate into each other's paths or off the edge of the plate (1 cm is ideal). A capillary tube is used for spotting. A small, tight spot is important for best results. The spotting solvent shall be volatile and relatively non-polar to minimize the wandering of the initial spot. The spot shall be dried (evaporated or heat dried) prior to entering the mobile phase of the test. Appropriately label the plate before spotting.

5.4.10.2.2. A developing chamber utilizing the glass tank apparatus and appropriate solvent(s) should be assembled and allowed to equilibrate before adding the spotted plate. The solvent of choice for the mobile phase will vary depending upon the compounds of interest. If the target compound or group of compounds is known, a good reference book (e.g. Clarke) will outline a TLC system that works for the compound.

5.4.10.2.3. Place the spotted TLC plate into the chamber in a vertical position with the origin line at the bottom but above the mobile phase solvent. The solvent should be allowed to migrate up the length of the plate. Attempt to remove the plate prior to the solvent reaching the top; spots may be lost if plate stays in the tank too long. A shorter migration distance increases sensitivity (the sample is concentrated), but decreases resolution. After the solvent has traveled the required distance, the plate is removed. The plate is allowed to dry by evaporation or heated with warm air. The plate is placed in a ventilated area and sprayed with an appropriate visualizing compound (depending upon the compounds screened for). Any developed spots are circled immediately if they are not clearly visible.

5.4.10.3. Interpretations

5.4.10.3.1. Compare results of unknown to those of standard. A spot shall match color and location (in-line with) of the standard for a positive preliminary result. The line of spots across the TLC plate should form a continuum, be it linear or parabolic.

5.4.10.3.2. Document the plate for the case file.

5.4.10.4. Controls

5.4.10.4.1. A blank and an authenticated drug standard(s) shall be run with each plate.

5.4.10.4.1.1. A blank TLC shall have no spot in the "blank" column.

5.4.10.4.2. The results of the controls shall be documented in the case notes.

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5.4.11. Marijuana

This procedure is composed of a series of tests, none of which by themselves are specific for marijuana or THC, but taken in combination, are considered specific for the presence of marijuana or its resins.

5.4.11.1. Supplies (equipment/reagents/standards)

5.4.11.1.1. Stereomicroscope.

5.4.11.1.2. Thin layer chromatography tank and silica gel plates.

5.4.11.1.3. Aerosol spray units, capillary tubes, test tubes.

5.4.11.1.4. Aqueous Fast Blue BB solution, or other appropriate visualizing spray.

5.4.11.1.5. Petroleum ether, methanol, chloroform (stabilized with ethanol or pentene), ethyl acetate, acetonitrile, ether or other appropriate solvent.

5.4.11.2. Method

5.4.11.2.1. Place plant material/residue in test tube and cover with appropriate solvent, or flush smoking device with appropriate solvent.

5.4.11.2.2. Use this extract for thin layer chromatography and modified Duquenois-Levine.

5.4.11.2.3. Retain small amount of unused solvent as blank.

5.4.11.2.4. Plant material is examined using a stereomicroscope for the following characteristics:

5.4.11.2.4.1. Cystolithic hairs-Small "bear claw" shaped hairs with bases of calcium carbonate. The cystolithic hairs are located on the topside of the leaf or leaf-fragment.

5.4.11.2.4.2. Unicellular hairs- Fine hairs located on the underside of the leaf or leaf-fragment. Unicellular hairs are not always observed on the leaves from the budding parts of the marijuana plant.

5.4.11.2.4.3. Seeds are examined using a stereomicroscope for the following characteristics:

- Veined shell.
- Ridged edges.
- Point on one end and dint on the end of plant attachment.

5.4.11.2.5. Modified Duquenois-Levine Test

5.4.11.2.5.1. In a test tube containing a portion of the evaporated solvent extract, mix 2-10 drops of Duquenois reagent and an equal amount of concentrated HCl.

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5.4.11.2.5.2. Let stand ½ to 3 minutes and observe color change.

5.4.11.2.5.3. Add chloroform.

5.4.11.2.5.4. Observe if purple color transfers into chloroform layer.

5.4.11.2.5.5. Record results in the case file.

5.4.11.2.6. Thin Layer Chromatography

5.4.11.2.6.1. Spot a small amount of solvent extract onto a TLC plate alongside of a marijuana standard and solvent blank.

5.4.11.2.6.2. Develop the plate using one or more of the following mobile phases:

- Chloroform
- Ethyl Acetate
- 4:1 Petroleum Ether/Ether

5.4.11.2.6.3. Visualize by spraying the plate with Fast Blue BB salt solution or other acceptable visualizing spray.

5.4.11.2.6.4. Compare results of unknown to those of standard. Document the plate for the case file.

5.4.11.2.7. Marijuana may also be confirmed on the GCMS, as long as the identification criteria are met per 5.4.4.

5.4.11.3. Interpretations

5.4.11.3.1. A positive test shall be defined as the following:

5.4.11.3.1.1. Microscopic - Observation of cystolithic hairs on the leaf and/or the presence of characteristic seeds.

5.4.11.3.1.2. Thin Layer Chromatography - Presence of a red spot with migration distance consistent with the red THC spot of the standard. The line of spots across the TLC plate should form a continuum, be it linear or parabolic. The blank shall be negative (no spot).

5.4.11.3.1.3. Modified Duquenois-Levine - A purple color developing after the addition of the HCl (color may vary from blue to reddish purple depending on the sample). Transfer of the color into the organic layer after the addition of chloroform.

5.4.11.3.2. A positive result shall be defined as the following:

5.4.11.3.2.1. Positive microscopic, positive single TLC system and positive modified Duquenois-Levine.

5.4.11.3.2.2. Negative microscope, positive modified Duquenois-Levine and two positive TLC systems.

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5.4.11.3.3. Marijuana shall be reported using the words "marijuana or the resins thereof."

5.4.11.4. Controls

5.4.11.4.1. A blank and a standard shall be run with each batch of Duquenois-Levine color tests and TLC plate(s). The blank (negative control) is the extraction solvent. The standard (positive control) is an authenticated marijuana standard.

5.4.11.4.2. The results of the controls shall be documented in the case notes.

5.4.12. Psilocybin Mushrooms

5.4.12.1. Supplies (equipment/reagents/standards)

5.4.12.1.1. GCMS and appropriate analytical software.

5.4.12.1.2. TLC equipment.

5.4.12.1.3. Methanol, acetone, or other appropriate solvents.

5.4.12.1.4. Fast Blue BB, p-Dimethylaminobenzaldehyde (pDMAB), or other appropriate solvents and/or visualizing sprays.

5.4.12.1.5. Ammonium hydroxide (NH₄OH).

5.4.12.1.6. Spot plates, pipettes, test tubes and caps.

5.4.12.1.7. Freezer.

5.4.12.2. Method

5.4.12.2.1. Weber Color Test

5.4.12.2.1.1. Add Fast Blue BB to spot plate. Ensure no color develops. Add sample. A positive test will turn red-orange within a couple of minutes.

5.4.12.2.1.2. Transfer some of the liquid to another clean well and add a drop of concentrated HCl. A positive test will turn a blue-green color.

5.4.12.2.2. GCMS Sample Preparation and Analysis

5.4.12.2.2.1. There are multiple extractions that can be performed on mushrooms to determine if they contain psilocybin and/or psilocin. The matrix of the sample may determine the appropriate extraction to use. Listed below are a couple of extraction procedures. However, it is up to the analyst's discretion to determine the appropriate extraction method to use based on their training and experience with different types of extractions.

5.4.12.2.2.2. Extract with just enough methanol to cover the sample in a test tube. At this stage, the extract can be injected into the GCMS.

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5.4.12.2.2.3. Centrifuge and decant solution into a clean test tube. Cap and place into freezer for at least one hour.

5.4.12.2.2.4. Remove from freezer and immediately add an equal volume of acetone, mix.

5.4.12.2.2.5. Centrifuge, decant, and if necessary, concentrate the supernatant.

5.4.12.2.2.6. Run on GCMS, using a split or splitless data acquisition method depending on the sensitivity of the instrument.

5.4.12.2.3. Thin Layer Chromatography

5.4.12.2.3.1. If differentiation of psilocyn and psilocybin is required, run a T1 system (10 mLs methanol + 7 drops of ammonium hydroxide (NH₄OH), developed with pDMAB.

5.4.12.3. Interpretations

5.4.12.3.1. Compare the results with an authenticated psilocyn or psilocybin standard. Psilocybin breaks down into psilocyn in the hot injection port of the GC.

5.4.12.3.2. Positive results can be reported as "psilocyn and/or psilocybin".

5.4.12.4. Controls

5.4.12.4.1. Negative and positive controls shall be run with the color tests.

5.4.12.4.1.1. An authenticated psilocyn/psilocybin standard shall be used as a positive control.

5.4.12.4.1.2. A clean well shall be used as a negative control.

5.4.12.4.2. The results of the controls shall be documented in the case notes.

5.4.13. LSD

5.4.13.1. Supplies (equipment/reagents/standards)

5.4.13.1.1. GCMS and appropriate analytical software.

5.4.13.1.2. Ultraviolet (UV) light source.

5.4.13.1.3. TLC equipment.

5.4.13.1.4. Methanol, chloroform, or other appropriate solvents.

5.4.13.1.5. Sodium carbonate, sodium bicarbonate, sodium hydroxide, or appropriate basic salt/solution.

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5.4.13.2. Method

5.4.13.2.1. Preliminary test

5.4.13.2.1.1. Place evidence under the UV light. LSD exhibits specific (blue-violet) fluorescence in the presence of ultraviolet light.

5.4.13.2.1.2. It is common for white paper to reflect the UV and appear violet even without LSD. If the entire sample fluoresces, (no differentiation from background and suspected location of LSD), then a methanol extract can be performed and then observed under UV light.

5.4.13.2.2. GCMS Sample Preparation and Analysis

5.4.13.2.2.1. "Window panes", blotter paper and pulverized microdots can be extracted directly with methanol. Place sample in test tube and add just enough methanol to cover the sample. Shake and let soak. Time can vary but they may need to soak for at least an hour. Centrifuge, if necessary, and analyze.

5.4.13.2.2.2. Sugar cubes, "SweetTarts" or other candy. View sample under UV to find the area of suspected LSD. Scrape off upper layer (attempt to reserve half of the sample). Dissolve sample into water and make basic. Extract with chloroform and run on the GCMS.

5.4.13.2.2.3. The GCMS should be set to splitless mode. The liner may have to be changed to a splitless liner depending on the sensitivity of the instrument.

5.4.13.3. Interpretations

5.4.13.3.1. Compare the results with an authenticated LSD standard.

5.4.13.3.2. Positive results shall be reported as "lysergic acid diethylamide (LSD, Schedule I)"

5.4.13.4. Cautions

5.4.13.4.1. The retention time is concentration dependent.

5.4.13.5. Controls

5.4.13.5.1. If a color test is utilized, a negative and positive control shall be run and documented in the case notes.

5.4.13.5.1.1. An authenticated LSD standard shall be used as a positive control. A purple color will develop if using pDMAB.

5.4.13.5.1.2. A clean well shall be used as a negative control.

5.4.14. GHB

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GHB (gamma-hydroxybutyrate) is a controlled substance in Idaho, while its precursors GBL (gamma-butyrolactone) and 1,4-Butanediol (BD) are not. This is problematic in that the interconversion of GBL to GHB and BD to GHB is simply pH dependent. In aqueous solutions, GHB and GBL will exist in equilibrium. The relative concentrations of each are also pH dependent.

5.4.14.1. Supplies (equipment/reagents/standards)

5.4.14.1.1. GCMS and appropriate analytical software.

5.4.14.1.2. pH paper.

5.4.14.1.3. Chloroform, methanol, ethanol, ethyl acetate or other appropriate solvents.

5.4.14.1.4. H₂SO₄, Bis(trimethylsilyl)trifluoroacetamide with trimethylchlorosilane (BSTFA with 1% TMCS, a trimethylsilyl (TMS) derivatizing reagent).

5.4.14.1.5. Bromocresol green, methyl orange, dextrose, aniline hydrochloride, sodium hydroxide.

5.4.14.2. Method

5.4.14.2.1. Color Test

5.4.14.2.1.1. Bromocresol green – mix 0.03g in 100 mL of 4:1 methanol:water. Adjust pH to 7 with NaOH.

5.4.14.2.1.2. Methyl Orange – mix 0.01g of Methyl orange in 100 mL of methanol. Adjust pH to 7 with NaOH.

5.4.14.2.1.3. Modified Schweppes

- Solution A – mix 2.0g dextrose in 20 mL of water
- Solution B – mix 2.4g aniline hydrochloride in 20mL of ethanol.
- Mix Solution A & B and dilute to 80 mL with methanol.

5.4.14.2.1.4. Mix Bromocresol green solution with the Methyl orange solution in a 1:1 ratio. Add 3 parts of this combined solution to 1 part of the Schweppes reagent.

5.4.14.2.2. Physical Tests

5.4.14.2.2.1. Pure GBL and BD are viscous liquids at room temperature. BD will solidify when placed in a refrigerator (4°C) while GBL will not.

5.4.14.2.2.2. GBL is soluble in chloroform, BD is not.

5.4.14.2.3. GCMS

5.4.14.2.3.1. GHB cannot be analyzed directly on a GCMS as it will convert to GBL in the heated injection port. Add concentrated sulfuric acid to

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aqueous sample, extract with chloroform and analyze. If GBL is detected, the sample must be derivatized with BSTFA before injection.

5.4.14.2.3.2. GHB

- Extract aqueous sample with chloroform, discard chloroform.
- Dry down aqueous layer with nitrogen or dry air. Sample can be warmed to expedite drying as long as the temperature remains below 60° C.
- Once sample is completely dry, add 100-200 µL of BSTFA.
- Cap sample and heat at 60-70° C for 15-20 minutes.
- Add ethyl acetate and analyze.

5.4.14.2.3.3. GBL

- Extract with chloroform and analyze.

5.4.14.2.3.4. BD

- If pure BD is suspected, dilute with methanol and inject.
- In aqueous samples, if concentrations of BD are high enough, extract with chloroform.
- Dry down sample, add methanol and analyze.
- BD will derivatize with BSTFA (add 100-200 µL of BSTFA, cap sample and heat at 60-70° C for 15-20 minutes, add ethyl acetate and analyze).

5.4.14.3. Extraction scheme

5.4.14.3.1. Solids

5.4.14.3.1.1. Perform color test.

5.4.14.3.1.2. If negative, dissolve in methanol and analyze on GCMS.

5.4.14.3.1.3. If positive, skip to 5.4.14.3.1.5.

5.4.14.3.1.4. If GCMS is negative, analysis is complete.

5.4.14.3.1.5. If GCMS has GBL, derivatize original sample with BSTFA and analyze on GCMS or run sample on FTIR.

5.4.14.3.2. Clear, thick liquids

5.4.14.3.2.1. Place 1-5 mLs of sample in the freezer for 15 minutes. If it solidifies, extract with methanol and analyze on GCMS. If results indicate the presence of GBL, proceed to section 5.4.14.3.3.4.

5.4.14.3.2.2. If sample remains a liquid, go to section 5.4.14.3.3.

5.4.14.3.3. Aqueous samples

5.4.14.3.3.1. Perform color test.

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5.4.14.3.3.2. Acidify a portion of the sample with concentrated H₂SO₄ and extract with chloroform. Analyze the chloroform layer on the GCMS. If results are negative for GBL, proceed with section 5.4.14.3.3.3. If GBL is present, skip to section 5.4.14.3.3.4. Report BD, if found.

5.4.14.3.3.3. If results are negative, take a portion of the original sample and dry down with nitrogen or air and heat (below 60° C). Extract with methanol and analyze with GCMS.

5.4.14.3.3.4. Take a portion of the original sample and extract with chloroform. Analyze the chloroform layer on the GCMS. Report GBL, if found.

5.4.14.3.3.5. Take aqueous layer from 5.4.14.3.3.4 and analyze using 5.4.14.2.3.2 (starting from second bullet).

5.4.14.4. Interpretations

5.4.14.4.1. A positive color reaction for the presence of GHB will turn green.

5.4.14.5. Cautions

5.4.14.5.1. Aniline is acutely toxic, handle with care.

5.4.14.6. Controls

5.4.14.6.1. Negative and positive controls shall be run with the color test and documented in the case notes.

5.4.14.6.1.1. An authenticated GHB standard shall be used as a positive control.

5.4.14.6.1.2. A clean well shall be used as a negative control.

5.4.15. Other Controlled Substances

5.4.15.1. If a specific method is not spelled out for analysis, it is up to the discretion of the analyst to determine the appropriate extraction to confirm the identity of the substance. This includes, but is not limited to, the analysis of khat, steroids, synthetic cannabinoids, substituted cathinones, fentanyl, or any number of designer drugs encountered in the laboratory.

5.5. Equipment

5.5.1. Only suitable and properly operating equipment shall be employed in the drug chemistry lab.

5.5.1.1. Equipment includes:

5.5.1.1.1. Balances.

5.5.1.1.2. Stereomicroscopes.

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5.5.1.1.3. GCMS.

5.5.1.1.4. FTIR.

5.5.1.1.5. Ultraviolet light box/Alternate light source.

5.5.1.1.6. Stir plates, magnets.

5.5.1.1.7. Test tubes, test tube caps, pipettes, spatulas, spot plates.

5.5.2. The manufacturer's operation manual and other relevant documentation for each piece of equipment shall be readily available.

5.5.3. Each piece of equipment that produces a result that has a significant impact to the final result shall be uniquely identified by its serial number (if applicable) or other unique identifier.

5.5.3.1. Equipment includes balances, GCMS and FTIR.

5.5.4. Before being placed into service, equipment used for analysis shall be calibrated or checked to ensure proper performance.

5.5.4.1. Equipment includes balances, GCMS, and FTIR.

5.5.5. Equipment performance parameters should be routinely monitored and documented.

5.5.5.1. All maintenance is recorded in a logbook.

5.5.5.2. The many types of maintenance that can be performed on the instrumentation in the laboratory are beyond the scope of this document. General maintenance shall be performed on an "as needed" basis. Refer to the manufacturer's manuals for specific maintenance instructions.

5.5.6. Balances

5.5.6.1. An intermediate check shall be performed monthly on all balances, using ASTM Class 2 (or equivalent) weights, to ensure they continue to meet the required specifications. Intermediate checks shall also be performed if the balance has been moved, placed back into service, or purchased.

5.5.6.2. Before a check is performed, the pan should be clean and the balance level.

5.5.6.3. The weights used shall cover the weighing range of the balance and/or the expected weights of samples.

5.5.6.4. A log shall be kept of the following:

5.5.6.4.1. Balance ID.

5.5.6.4.2. Analyst initials.

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5.5.6.4.3. Date performed.

5.5.6.4.4. Weights used.

5.5.6.4.5. Recorded weight.

5.5.6.4.6. Whether results are within the tolerance of the balance.

5.5.6.5. Each balance has a tolerance range that shall be used to determine if the scale passes or fails the monthly check. This tolerance range shall be located on the balance check spreadsheet.

5.5.6.5.1. If a balance fails an intermediate check (falls outside the tolerance range), the check is repeated. If the balance still fails, the balance will immediately be taken out of service until it can be recalibrated or repaired. The balance shall clearly be labeled "Out of service".

5.5.7. Gas Chromatograph/Mass Spectrometer (GCMS)

GCMS is an instrument that combines the separation capabilities of Gas Chromatography with the compound selectivity of Mass Spectrometry. This instrument combination is used as a Category A technique in drug chemistry analysis. Separately, the GC and MS may be used as Category B tests.

5.5.7.1. Supplies (equipment/reagents/standards)

5.5.7.1.1. GCMS and computer with corresponding Agilent software.

5.5.7.1.2. Spectroscopic grade solvents or better, to include; methanol, chloroform, petroleum ether, acetone, acetonitrile, or other appropriate solvents.

5.5.7.1.3. Sodium carbonate, sodium bicarbonate, sodium hydroxide, or appropriate salt/solution.

5.5.7.1.4. Hydrochloric acid or other acid/acidic solution.

5.5.7.1.5. n-tridecane.

5.5.7.1.6. GCMS vials and caps.

5.5.7.1.7. Ultrapure Helium gas.

5.5.7.1.8. Authenticated drug standards.

5.5.7.1.8.1. A reference collection of primary and secondary standards that are used for comparisons shall be maintained by the lab.

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5.5.7.1.8.2. Analytical reference materials shall be made by diluting a small sample of a primary or secondary standard with an appropriate solvent or by performing a liquid/liquid extraction on the standard.

- The name of the standard, manufacturer and lot number (if applicable) shall be documented on the vial.
- These standard solutions shall be stored by the GCMS or in the freezer or refrigerator.

5.5.7.1.8.3. Primary and secondary standards may continue to be used for comparison purposes as long as they are of sufficient quality to ensure the integrity of the analytical results.

5.5.7.2. Instrument Tune

5.5.7.2.1. The MS tuning standard used is perfluorotributylamine (PFTBA) and is added directly to the appropriate storage vial on the mass spectrometer and filled as needed.

5.5.7.2.2. The PFTBA is maintained in a sealed and controlled environment within the instrument. Based upon years of laboratory experience, PFTBA has been shown to have an extensive shelf life that may extend well beyond manufacturer expiration dates. Therefore, a specific lot may remain in use beyond the labeled expiration as long as the instrument tune response appears normal. However, should an instrument fail a tune, one should consider PFTBA as a possible cause of the problem in their troubleshooting protocol.

5.5.7.2.3. The MS shall be tuned each day that the instrument is used for casework, using an AUTOTUNE (Atune.U).

5.5.7.2.4. An AUTOTUNE shall be run after every major maintenance procedure, i.e. source cleaning or column change.

5.5.7.2.5. Successful tunes shall be initialed by a drug chemist and kept in a logbook.

5.5.7.2.6. Definition of a successful tune (using PFTBA)

5.5.7.2.6.1. Low water and air (18, 28, and 32 amu).

5.5.7.2.6.2. Mass assignments within ± 0.2 AMU of 69, 219, 502.

5.5.7.2.6.3. Peak widths (PW) should be consistent and be 0.6 ± 0.1 (PW50).

5.5.7.2.6.4. 69 or 219 should be the base peak.

5.5.7.2.6.5. Base peak abundance should be $\geq 400,000$ but $\leq 600,000$.

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5.5.7.2.6.6. Relative abundances should be anything greater than 30% for 219 and anything higher than 1% for 502.

5.5.7.2.6.7. Isotope mass assignments should be ~1 AMU greater than the parent peak and the ratios should be 0.5-1.5% for mass 70, 2-8% for mass 220, and 5-15% for mass 503.

5.5.7.3. Method

5.5.7.3.1. A capillary column and temperature program shall be used which are capable of resolving the compound(s) in question.

5.5.7.3.2. Samples for GCMS analysis shall be diluted or extracted with solvents.

5.5.7.3.2.1. Once extracted, the sample extract is not considered evidence and does not need to be treated as such.

5.5.7.3.2.2. If the sample extract is retained, it shall be sealed inside the evidence packaging and returned with the evidence. This shall be noted on the report.

5.5.7.3.3. Samples may be injected manually or via an auto-sampler.

5.5.7.4. Interpretation

5.5.7.4.1. Identification of unknowns shall be determined by comparing the overall peak shape, baseline resolution, and MS spectrum of the sample unknown to the drug standard. In general, peaks should be roughly symmetrical and resolved to baseline. However, it is understood that some compounds and matrices may preclude such criteria; the analyst should be able to articulate why their data still meets quality standards (e.g., certain compounds always tail and some closely eluting peaks "shoulders" can still have readily identifiable mass spectra).

5.5.7.4.2. The difference between the GC retention times of the sample and standard shall not exceed 0.040 minutes. For this purpose, the retention times compared will be those shown in the total ion chromatogram (TIC) window.

5.5.7.4.3. Chromatography peaks should be of sufficient abundance to produce an acceptable mass spectrum.

5.5.7.4.4. For a mass spectrum to be acceptable for identification:

5.5.7.4.4.1. The relative intensities of the major peaks should agree within 20% between the reference and the sample spectrum.

5.5.7.4.4.2. All ions with a relative intensity greater than 10% of the base peak in the reference spectrum must be present in the sample spectrum. For those compounds that do not have three major ions above 10% relative abundance (e.g., methamphetamine, methadone),

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at least 8 ions from the drug standard should also be present in the sample.

5.5.7.4.4.3. The base peak of both the reference spectrum and the sample must be the same, except for those compounds where other ions are present with relative intensities within 20% of the base peak. Under those circumstances, shifting of the base peak is acceptable as long as the relative intensities of the major peaks agree within 20% between the reference and sample spectra (i.e. cocaine).

5.5.7.4.4.4. Any ions present in the sample spectrum but not in the reference spectrum should be reviewed for the presence of contamination or the presence of co-eluting compounds.

5.5.7.5. Controls

5.5.7.5.1. Method and instrument blanks shall be utilized to check for contamination or carryover issues. A method blank consists of all solvents and reagents that the sample is exposed to while analyzing. Any appropriate solvent can be used as an instrument blank.

5.5.7.5.2. Each day the instrument is used for casework, and prior to casework being performed, an instrument blank (negative control) and an authenticated drug standard (positive control) shall be run.

5.5.7.5.3. If the tune passes, no contamination is identified that could impact casework, and the known standard performs like it should, then the instrument is clear for use.

5.5.7.5.4. Because this is a known standard (positive control), the presence of stray peaks may indicate breakdown products or contamination. If stray peaks are considered significant or expected peaks are not of sufficient abundance, the problem shall be evaluated. This may require instrument maintenance or the vial to be discarded and a fresh vial used.

5.5.7.5.5. A method blank shall be run immediately before each sample. It is considered blank if the compound(s) of interest would not be identified using criteria in 5.5.7.4.

5.5.7.5.5.1. If running a sample for investigative purposes only (see 5.4.4.14), method blanks are not required.

5.5.7.5.6. The presence of extraneous peaks in the blank chromatogram may indicate potential contamination. If a blank has an identifiable compound of interest, then the blank shall be rerun to determine if it is contaminated. If it still contains an analyte(s) of interest, it shall be discarded and a fresh vial used.

5.5.7.5.6.1. The sample shall be rerun immediately after the acceptable blank (Ensure that no solvents or reagents have been changed prior to

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making a new method blank. If this has occurred, the sample shall be re-extracted with the current solvents/reagents).

5.5.7.5.6.2. Once a month, and after maintenance, a test mix, consisting of a mix of common drugs or chemicals, shall be run. The chromatogram shall be compared to the previous month's run for response, separation and identification. Any discrepancies shall be noted and addressed (more maintenance or fresh vial), if necessary. The chromatogram shall be retained.

5.5.8. Fourier Transform Infrared Spectrophotometer (FTIR)

Infrared spectroscopy is a Category A technique in the analysis of substances. Samples can be liquid, solid or vapor, but they shall be relatively pure for purposes of identification.

5.5.8.1. Supplies (equipment/reagents/standards)

5.5.8.1.1. FTIR and corresponding analytical software.

5.5.8.1.2. IR grade potassium bromide (KBr).

5.5.8.1.3. Spectroscopic grade solvents or better, to include; methanol, chloroform, petroleum ether, acetone, acetonitrile, or other appropriate solvents.

5.5.8.1.4. Hydraulic or hand press, die set, gas cell apparatus, dessicator (if applicable).

5.5.8.1.5. Authenticated drug standards.

5.5.8.2. Method

5.5.8.2.1. Background spectra shall be collected immediately before every sample, under the same conditions as the sample.

5.5.8.2.2. Samples may be run utilizing the Attenuated Total Reflectance (ATR), pressed into a pellet with potassium bromide or analyzed with a vapor cell.

5.5.8.2.2.1. ATR preparation

- Place sample (solid or liquid) on crystal (i.e. diamond), press and analyze.

5.5.8.2.2.2. KBr pellet

- A small amount of KBr is placed in a mortar along with a small aliquot of sample.
- Some samples that are dissolved in a solvent (i.e. MeOH or petroleum ether) can be dried directly on KBr in a mortar before making the pellet.

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- Thoroughly ground the sample with a pestle until a fine powder is obtained.
- Transfer some of the mixture to a die set or IR card.
- Using a hydraulic or hand press, press the sample into a relatively transparent pellet.
- Place pellet in sample chamber and analyze.

5.5.8.2.2.3. Volatiles

- Volatile samples can be analyzed in a gas cell apparatus that contains clear KBr windows on each end and an opening for sample addition.

5.5.8.3. Interpretation

5.5.8.3.1. In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard in the internal library produced by ACSO.

5.5.8.3.1.1. An internal library shall be made using authenticated drug standards.

5.5.8.3.1.2. Sample and standard analysis shall be performed under like conditions (i.e. ATR vs KBr).

5.5.8.3.2. FTIR spectra are considered a match if the peaks of the standard are present in the sample, in location, shape and relative intensities. Any extra major peaks in the sample must be explainable.

5.5.8.3.3. If spectral subtraction is done, then what was subtracted shall be in the case notes.

5.5.8.3.4. If an FTIR spectrum is inconclusive or negative for a controlled substance, the sample shall then be analyzed on a GCMS.

5.5.8.4. Controls

5.5.8.4.1. An instrument performance verification shall be run monthly and after any maintenance. Using the manufacturer's procedures, a performance verification of the instrument is done using a traceable polystyrene film. The printouts shall be initialed and placed in a logbook.

5.5.8.4.1.1. If the verification does not pass and/or there is any other symptom of system failure, perform a bench alignment and/or consult the manufacturer.

5.5.8.4.2. A bench diagnostics test shall be run monthly and after any maintenance. The printouts shall be initialed and placed in a logbook.

5.5.8.4.2.1. If any of the bench diagnostics tests fail, perform a bench alignment and/or consult the manufacturer.

5.5.8.4.3. Check the desiccant monthly, and replace if necessary.

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5.6. Traceability

- 5.6.1.** Weights shall be NIST traceable and certified at time of purchase. The documentation for the certification/calibration of the weights shall be retained.
- 5.6.2.** Weights used to check the balance accuracy shall be re-certified every accreditation cycle by an ISO/IEC 17025:2005 accredited vendor whose scope of accreditation covers the calibration performed.
- 5.6.3.** For balances used in weighing evidence, the uncertainty of measurement (variability) of the balance has been determined by an in-house empirical performance study of that balance. The uncertainty of measurement (UM) is calculated annually and updated (if necessary). The current uncertainty values can be found in the electronic laboratory documents. The uncertainty values and windows are posted near the balances.
- 5.6.4.** Annually, balances shall be calibrated by an outside vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed.
- 5.6.5.** Case notes shall indicate the uncertainty value for the balance(s) used when the total weight of a sample(s) falls within the window of uncertainty at regulatory limits.
- 5.6.6. Uncertainty of Measurement Calculations**
- 5.6.6.1.** The “d” value of a balance is the readability of the balance or the smallest division displayed on the balance readout. The “d” value also shows the precision obtainable for the balance (e.g., precision to the 10ths, 100ths, or 1000ths).
- 5.6.6.2.** Regardless of what scale is used, uncertainty values not already in increments of “d” shall be rounded up to the nearest “d” increment. For example: If the uncertainty of a scale with d-value equal to 0.005 kg is 0.013 kg, then the uncertainty shall be rounded up to the nearest increment of “d” resulting in an uncertainty of 0.015 kg.
- 5.6.6.3.** The measurement result shall include the measured quantity value (X), along with the associated expanded uncertainty (U), and this measurement shall be reported as $X (\pm) U$, where U is consistent with the units of X.
- 5.6.6.3.1.** For a single measurement, the reported weight of an exhibit shall be the balance reading \pm the calculated uncertainty.

Example 1: (assume a d-value of 0.01):

The reading from the scale is 1.23g and the uncertainty value is 0.01.

The reported value shall be 1.23g \pm 0.01g.

5.6.6.3.2. For multiple measurements, one cannot simply add all of the uncertainties together and report the combined uncertainties as the total uncertainty. This is because the error is random; sometimes the measured weight will be higher and other times it will be lower than the true value.

5.6.6.3.3. To calculate the Uncertainty (U) for multiple measurements with the same uncertainty, Equation 1 must be applied.

Equation 1

$$U = \sqrt{N \times (u_b)^2}$$

U = total Uncertainty for the sum value
N = number of measurements
 u_b = Uncertainty of the balance

Equation 1 can be simplified to Equation 2.

Equation 2

$$U = \sqrt{N} \times u_b$$

Example 2:

A balance used to obtain the weight for five bindles has an uncertainty of 0.01 gram. Table 2 shows the weights of the 5 bindles using this balance.

Table 2

Bindle #	Weight (g)
1	0.52
2	0.67
3	0.78
4	1.02
5	0.21
Total	3.20 g

The uncertainty from each weighing shall be considered. To determine the combined uncertainty from all five weighings, use Equation 2.

Using Equation 2, the combined uncertainty would be calculated as follows:

$$U = \sqrt{N} \times u_b = \sqrt{5} \times 0.01 = 0.022 \text{ g}$$

Since uncertainty values will have the same precision as the balance being used and shall be rounded up to the nearest increment of "d" (d=0.01), the calculated total uncertainty value of 0.022 gram would be rounded up to 0.03 gram.

The final weight is then reported as 3.20 grams \pm 0.03 g.

Example 3 (significant figures and d-values):

A case has ten bags of marijuana, each weighed separately:

Total Weight: 52.015 kg

Scale Uncertainty: 0.005 kg

d-value: 0.005 kg

Using Equation 2, the combined uncertainty would be:

$$U = \sqrt{N} \times u_b = \sqrt{10} \times 0.005 = 0.0158 \text{ kg}$$

This combined uncertainty value would be rounded up to the precision of the scale (i.e., 0.016 kg) and then up to the nearest increment of "d", making the final uncertainty 0.020 kg.

The final weight shall be reported as 52.015 kg \pm 0.020 kg.

5.6.6.3.4. At times, individual items within an exhibit will necessitate using two different balances or different weights may fall within different calculated uncertainty values for a particular balance (see note below). The example below would apply to either of these situations. These calculations also apply to subtraction calculations; although the difference is found through subtraction, the combined uncertainty is the sum of the separate uncertainties. To calculate the Uncertainty (U) for multiple measurements with more than one U value, see example below.

Example 4:

A submission came into the laboratory that consisted of four plastic bags and three paper bags all containing material that was visually consistent. Due to the different sizes of the containers, the analyst decided to weigh the contents of the plastic bags on a 400-gram capacity balance with an uncertainty value of 0.01 gram and weigh the contents of the paper bags on a larger capacity balance with an uncertainty value of 0.4 gram.

As an aside, if the analyst made all the measurements on a single 600-gram capacity scale that had separate uncertainty values for different ranges of weights, the "d" value would be the same for both weight levels.

Table 3: Balance B1

Plastic bags	Weight (g)
1	10.56
2	14.66
3	28.50
4	31.27
Total	84.99 g

Uncertainty = 0.01 g, "d" = 0.01

Table 4: Balance B2

Paper bags	Weight (g)
1	450.6
2	422.8
3	575.2
Total	1448.6 g

Uncertainty = 0.4 g, "d" =0.2

For this calculation Equation 2 shall be used twice – once for B1 and once for B2. The combined equation (Equation 3) will be as follows:

Equation 3

$$U_{tot} = \left(\sqrt{N_{B1}} \times u_{B1} \right) + \left(\sqrt{N_{B2}} \times u_{B2} \right)$$

U_{tot} : total uncertainty
 N_{B1} : number of measurements on Balance 1
 u_{B1} : uncertainty of Balance 1
 N_{B2} : number of measurements on Balance 2
 u_{B2} : uncertainty of Balance 2
 u_{B1} : uncertainty of Balance 1

Using the above numbers for the weights obtained in Table 3 and Table 4, the following measurement of uncertainty is obtained.

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$$U_{tot} = (\sqrt{4} \times 0.01) + (\sqrt{3} \times 0.4) = 0.71 \text{ g}$$

The combined net weight (total of balance B1 plus total of balance B2) is 1533.59 grams. When combining measurements with different degrees of precision, *the precision of the final answer can be no greater than the least precise measurement*. With this rule in mind, the total weight would be 1533.5 grams (one decimal place). The weight itself is never rounded up, but rather is truncated. The calculated uncertainty (U) equaled 0.71 gram, which would be rounded up to 0.8 gram (uncertainty sum rounded up to the nearest increment of the larger "d" value). The reported net weight shall be 1533.5 g ± 0.8 g.

5.6.7. Reference materials

5.6.7.1. In drug chemistry, the reference materials are the drug standards (primary or secondary) that are used as a reference for confirmatory analysis.

5.6.7.2. Standards shall be obtained from commercial or government sources (primary standards) or from previously analyzed casework (secondary standards).

5.6.7.3. Primary standards shall be uniquely identified by the manufacturer and lot number. For secondary standards, the case number (and exhibit number, if applicable) shall be used as the lot number.

5.6.7.4. All drug standards shall be inventoried. A Drug Standard Form shall be filled out for all standards that have more than a one-time use.

5.6.7.4.1. The form shall list the drug, schedule, source, initial gross weight, current weight and date of authentication.

5.6.7.5. Annually, the standards shall be inventoried. This shall be documented on the Drug Standard Form.

5.6.7.6. All standards shall be securely stored in the laboratory in a double locking key box. Access to the standards shall be limited to personnel designated by the laboratory manager. Two designated personnel are required to open this box at any given time.

5.6.7.7. A logbook shall be maintained for the standards double locking key box that lists the date and initials of personnel accessing it.

5.6.7.8. Documentation for the standards shall also include the DEA Form-222 and receipt of purchase.

5.7. Sampling

5.7.1. The principal purpose of sampling is to allow the analyst to draw a conclusion about the entire population by examining only a portion of the population (i.e., a sample). If a scientist is only analyzing a single item or drawing a conclusion

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regarding only the items tested with no inference to the entire population, then a sampling plan is not needed. If no sampling plan is needed then analysis can be accomplished in one of two ways:

- 5.7.1.1.** Itemize the examined unit (or multiple units) separately from the non-examined units. The report would then have a finding associated with the analyzed unit(s) and a "Not analyzed" finding associated with the non-examined unit(s).
- 5.7.1.2.** Keep the population whole (i.e. not itemized out), but fully examine one unit. The report would then clearly convey that the finding is attributed to only one unit and the rest of the units were not analyzed.
 - 5.7.1.2.1.** Example: "20 white oblong tablets with imprint "M367", analyzed one. The sample contains Hydrocodone (CII)."
- 5.7.1.3.** With a statistical sampling plan the analyst can test a sample of a visually-consistent population and report a result that applies to the entire population with a stated level of confidence. The statistical sampling plan utilized shall be the hypergeometric sampling plan. Refer to the Hypergeometric Table (Table 5). This table was constructed using the hypergeometric calculation based on a 95% probability that 90% of the population contains the identified compound. For an in-depth explanation of this calculation, click on the link provided.
https://www.unodc.org/documents/scientific/Drug_Sampling.pdf
 - 5.7.1.3.1.** The case notes shall clearly indicate if a hypergeometric sampling plan was used.
 - 5.7.1.3.2.** An observation shall be conducted to determine that one homogenous population is present. If more than one population is present, the sampling plan shall be used on each type of population.
 - 5.7.1.3.3.** The analyst shall include in their notes the population size and the number to be tested. See example below.
 - 5.7.1.3.3.1.** Hypergeometric Sampling Plan, Population size = 40
Number of samples to be tested =20
 - 5.7.1.3.4.** The appropriate number of specimens within the population will be randomly selected.
 - 5.7.1.3.5.** The notes shall indicate the corresponding confidence limits (i.e., "95% confident that at least 90% of the items in the population contain the reported drug").

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Table 5: Hypergeometric Table

Total number of items in exhibit	Number of units to be tested
1-10	All units
11 – 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

5.8. Handling of test items See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual.

5.9. Assuring the quality See Ada County Sheriff's Office Forensic Lab Quality Assurance Manual

5.10. Reporting

5.10.1. In addition to the reporting requirements outlined in the Quality Manual, a controlled substance report shall also contain the original weight or number of plants, pills/capsules, and the schedule of the drug, if applicable (weight/volume of liquids is not reported).

5.10.2. For any weight that registers 0.09g or less on the balance, it shall be reported as "< 0.10g".

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- 5.10.3.** If the sample is not weighed (and can't be counted), due to a small amount, the report shall have "residue" in the description of the sample.
- 5.10.4.** If an extract(s) must be returned with the evidence (per 5.4.6.1 and 5.4.6.2) the report shall state "**Remaining extract(s) was/were returned with evidence.**"
- 5.10.5.** If uncertainty of measurement is reported, the measurement result shall include the measured quantity value (X) along with the associated expanded uncertainty (U) and shall be reported as X +/- U, where U is consistent with the units of X. (Example: 28.05g +/- 0.04g).
- 5.10.6.** If uncertainty of measurement is reported, the report shall also state "**The uncertainty was calculated at the 95% confidence level**".
- 5.10.7.** If a substance cannot be identified after meeting the minimum examination requirements than "**No controlled substance(s) detected**" shall be reported.
- 5.10.8.** If a controlled substance is present but not confirmed, the report shall read "**Preliminary results indicate the presence of _____, not confirmed**".
- 5.10.8.1.** The compound and/or class of compound shall be reported.
- 5.10.8.2.** The reason why the substance was not confirmed shall be on the report.
- 5.10.8.2.1. Exception:** When a sample is brought into the lab to be tested for a preliminary result only, a "Preliminary Results" report shall be issued. It is not necessary to include a reason why it was not confirmed.
- 5.10.9.** Non-analytical identifications of pills shall read, "**Source lists as _____**". If the relative amounts of the therapeutic ingredients will affect the scheduling, than they shall be recorded on the report.
- 5.10.10.** If there is limited sample to test during the course of the examination and/or the analyst is unable to satisfy the minimum examination requirements than "**Insufficient sample for identification**" shall be reported.
- 5.10.11.** For synthetic cannabinoids that have ambiguous scheduling, the report shall read, "**_____, a synthetic cannabinoid.**"

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Drug Chemistry Analytical Method History

Issuing Authority: Quality Assurance Manger

SECTION & COMMENTS	DATE ADOPTED	AUTHOR	REVIEWER(S)
<p>Version 2, 5.4.7-5.4.15 changed Table of Contents titles</p> <p>3.0 added internal standard and derivatization to definitions</p> <p>4.6.1 added critical supplies</p> <p>5.1.1.4 exception to Duq-Lev bulk reagent added 5.3 section</p> <p>5.4.4.10 added the word "ran"</p> <p>5.4.4.14 reworded how we handle hand-delivered samples</p> <p>5.4.9.1.2 CHCl₃ and UV light added to color tests</p> <p>5.4.10.3 added TLC line can form a continuum</p> <p>5.4.11.1.5 added "or pentene"</p> <p>5.4.11.2.5 added "modified" to test</p> <p>5.4.11.3.2.1 added the word positive</p> <p>5.4.12.3.1 changed wording from verified to authenticated</p> <p>5.4.13.2.2.1 changed time allotment</p> <p>5.4.13.4 switched order of "Cautions" and "Controls" to be consistent with other sections</p> <p>5.4.14 updated numbering issues</p> <p>5.5.1.1.7 added "spot plates"</p> <p>5.5.6.1 changed wording from "better" to "equivalent"</p> <p>5.5.7.1.8.2 changed wording from reference standards to reference materials and added refrigerator to storage location for drug std solutions</p> <p>added 5.5.7.5.5.1 not necessary to run blanks before presumptive screening tests on GCMS</p> <p>5.7.1.3 corrected site address and added to sampling;</p>		HC	

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