

SFPD Crime Lab Controlled Substances SOP

Version 06/23/05

REV.5

Approved: MB

**San Francisco Police Department
Criminalistics Laboratory**

Controlled Substances SOP

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1. GENERAL INFORMATION

A. Mission Statement

To provide thorough, accurate and timely examination of narcotics evidence for any public agency in the City and County of San Francisco (for other public agencies as time permits) and to give expert testimony in criminal and civil proceedings.

B. Scope of Work

1. This laboratory examines solids (e.g. crystals, tablets, amorphous materials), powders and liquids for the presence of controlled substances. Generally, a written request for the analysis of controlled substances is not required.
2. The terms “Narcotics” and “Controlled Substances” are interchangeable in this SOP.
3. Presumptive and confirmatory tests are routinely performed for most submissions and are described in the sections that follow.
4. Quantitative analyses are not conducted.
5. In addition to testing evidence, the narcotics analysis section maintains records of the examinations and reports the results of the analyses to the department.
6. Analysts provide expert testimony in a court of law.
7. Narcotics staff provides instruction to law enforcement personnel and other members of the criminal justice system in the areas of collection, preservation and submission of physical evidence, as well as capabilities of the Narcotics Analysis Section of the laboratory.

C. Authorized Personnel

1. Once Narcotics evidence is delivered to Hunter’s Point, only personnel assigned to the Criminalistics Laboratory are authorized to handle controlled substance evidence.

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2. Non-laboratory personnel must be accompanied by a member of the laboratory staff in the drug analysis section.

D. Safety

1. The SFPD Criminalistics laboratory safety manual covers procedures for safe laboratory practices. A Criminalist from each operational unit is assigned to a safety committee. Any safety committee member can be consulted on a variety of safety-related matters.
2. One goal of the laboratory is to promote an accident-free work environment. This is accomplished by providing periodic training, raising chemical awareness through education, making informational resources available and encouraging suggestions for improvements.

2. EVIDENCE INTEGRITY

A. Introduction

It is a fundamental goal to handle evidence in a manner that does not compromise its integrity. This includes ensuring that it is received in a sealed envelope, maintaining an obvious chain-of-custody, preventing loss and contamination, attempting to retain enough sample for reanalysis and securing it in the laboratory at all times.

B. Evidence Submission (See Section 3 for Chain of Custody)

1. Narcotics evidence from the field is generally sealed in an “Analyzed Evidence” envelope (SFPD PR. 7.0.12) and is assigned a laboratory number through the SFPD CABLE (Computer Assisted Bay-Area Law Enforcement) system. Officers record case and exhibit identifying information on the envelopes.
2. When an SFPD PR. 7.0.12 evidence envelope is too small to contain the exhibits, they can be sealed in a suitable container (e.g. cardboard box or paper bag). An “Analyzed Evidence” envelope, with case and exhibit information recorded, must be securely attached to the container.
3. Normally, narcotics evidence is submitted to the Property Control Division (PCD) at the Hall of Justice via a Drop Box. During the work week, a courier assigned to PCD transfers the narcotics evidence to Building 606 Crime Lab. There are two exceptions to this procedure:

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- a. A sealed evidence envelope or a larger suitably sealed submission may be delivered directly to the Crime Lab during business hours by an officer or other authorized person.
 - b. A member of the Crime Lab staff may receive evidence directly at another location, such as the Hall of Justice.
4. The submission guidelines for controlled substances (from field recovery) are as follows:
- a. Exhibits should be parceled in separate containers (e.g. ziplocks) before placing into the envelope.
 - b. If exhibits have been assigned numbers in a report before submission to the laboratory, they should be indicated on the envelope.
 - 1) If no numbers are present, the analyst may assign one.
 - 2) The analyst may rely on evidence descriptors (e.g. weight or number of units) to self-indicate each item.
 - c. All evidence envelopes must be sealed. This means that entrances into the envelope are taped, glued or wax-sealed. The seal should also bear the sealer's initials and/or star number.
 - 1) Stapled packages must also be taped or wax-sealed in order to be considered as "received in a sealed condition".
 - 2) Occasionally, a submission is adequately sealed (that is, prevention of loss or cross-contamination has been addressed and no unknown entry to the evidence could have been made), but there are no initials or star number on the seals. In this case, the analyst may add his or her initials to tape added to cover the seal.
 - 3) If the receiving analyst adds a tape seal or initials, the action will be indicated on the narcotics record form (Section 3.F.1).
 - 4) For large cases submitted in packaging other than a narcotics envelope (e.g. large plastic bags of marijuana plants), the integrity of the existing seal is analyzed. Notation of sealing

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inadequate for long-term storage is made on the record form. Repackaging may be necessary and will be indicated.

- 5) If a package lacks an adequate seal, it will be refused. A call to the officer or unit supervisor will be made. Evidence may be resubmitted once it is properly sealed.
- d. The charges against the individual should be listed.
 - 1) 11350, 11351, 11352 and 11360 charges for pharmaceutical tablets should be clearly indicated.
 - 2) An 11357b violation for suspected marijuana must be made clear.
 - 3) "Pending" is a suitable designation, if applicable.
- e. All special instructions must be clarified.
 - 1) Items slated for destruction must be clearly marked.
 - 2) Residues that are critical to a case should be indicated and contact information should be written on the envelope.
- f. Saliva-, blood- or feces-contaminated evidence should be indicated with a yellow biohazard label affixed to the outside of the evidence envelope. At a minimum "biohazard" should be written on the envelope in the special instructions section and a notation as to where on the body the evidence was obtained should be made.
- g. If a syringe is submitted, it must be placed in a rigid, shatterproof container before placing in the envelope. A biohazard label and indication of syringe must be made clear. It is laboratory policy to **NOT** routinely test the contents of syringes, though special circumstances may warrant it after a consultation with the Narcotics Supervisor.
- h. An accurate description of the contents is extremely useful to laboratory personnel, e.g. green leafy substance, possible peyote buttons or suspected dried mushroom are each better than solely "plant material".
- i. Officers submitting cases consisting of several bags or envelopes will indicate on each envelope which envelope of how many it is (e.g. "3 of 4 packages" or "3/4").
- j. Bulk items are to be properly sealed and have a completed evidence envelope attached to each package.

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- 1) Large marijuana seizure submissions must have each bag clearly labeled. Roots and planting containers should be discarded before submission. If present, they will be discarded in the Crime Lab.
- 2) Additionally, plants cannot be submitted drenched with water. They must be surface-dried, otherwise molding will occur. The plant material itself will be further dried in the laboratory.

C. Chain of Custody

1. Every evidence envelope, bag or box that is received through the Drop or directly by the Property Control Division (PCD) will be initialed, dated (time-stamp is acceptable) and checked for packaging condition by the individual receiving evidence. In instances where evidence is submitted unsealed, the person receiving it will contact the officer submitting the evidence. Only after proper sealing will the evidence be accepted.
2. All controlled substance evidence should have an assigned Laboratory Number when received. If there is no Laboratory Number, one must be obtained from the SFPD CABLE computer system.
3. Generally, a PCD member logs case information into SFPD CABLE at the PCD location. On occasion, a PCD member transports properly sealed evidence to the Crime Lab and a member of the Crime Lab staff logs the cases into CABLE.
4. Bar-coding of Evidence.
 - a. Each PCD member handling controlled substances evidence, and all Crime Lab staff members, have unique personal passwords that are required to gain access to the bar-code program. Personnel must log-off the program upon completion of entering one or more chain of custody transaction(s).
 - b. A PCD member attaches a bar-code label to the envelope and logs case information into the Narcotics database. Each package of evidence (envelope, bag or box) will have a unique bar-code, thus one case may have several associated bar-coded exhibits.

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- c. Narcotics evidence is then transported to Hunter's Point Building 606 by an SFPD officer. All evidence is transferred from PCD possession to Crime Lab storage area possession by bar-code scanning, in an electronically secure manner, by use of passwords.
 - d. All intra-lab evidence transfers between analysts are recorded in the database by scanning the bar-code in an electronically secure manner, by use of passwords.
 - e. Before the return trip to the Hall of Justice from Hunter's Point, previously analyzed, reviewed and sealed envelopes are scanned back to PCD possession in an electronically secure manner, by use of passwords. The bar-code transfer summary for any case may be printed as necessary.
5. Evidence may be destroyed pursuant to a properly executed court order. This destruction is done in conjunction with the PCD and the District Attorney's office. Destroyed evidence is indicated in the CABLE computer system and bar-code database.

D. Evidence Security at the Crime Lab

1. The Narcotics Analysis Laboratory must be locked when no analysts are present.
2. Any visitors, police officers not assigned to the laboratory, janitors or maintenance personnel must be accompanied by a crime lab staff member while in the Narcotics Analysis Laboratory.
3. Sealed, bar-coded evidence is delivered from the Hall of Justice to a designated, secure storage locker in the Narcotics Analysis Laboratory. If no analysts are present, this laboratory will be locked.
4. Cases are divided among the analysts working a given shift and transfer of custody is scanned in an electronically secure manner, by use of passwords.
5. While a case is open, the envelope and packaging remain in the analyst's workspace. Samples of the exhibits may go to common spaces of the lab (e.g. hoods, GC-MS station, stereomicroscope). The analyst may leave a case open in his or her workspace while taking short breaks. During long breaks (>1 hour) exhibits are to be repackaged and kept under staple-seal in the evidence envelope. At the end of the workday, cases (completed or incomplete) must be locked in the analyst's storage locker in the narcotics analysis laboratory.
6. Keys to the lockers are under the control of the individual assigned to the locker and the narcotics supervisor.

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7. One case per analyst is allowed to be open at a time. After analysis, the evidence envelope is staple-sealed and another case may be opened. While awaiting review, the staple-sealed envelopes are stored in a locker.
8. After analysis and review of cases, re-sealed and initialed evidence is scanned back to secure Crime Lab storage area location in an electronically secure manner, by use of passwords, and placed in the return evidence box. This box remains secured in the Narcotics Analysis Laboratory at all times.

E. Controlled Substance Standards Security

The drug specimen lockers are to remain locked at all times unless an analyst is present in the room.

3. TECHNICAL PROCEDURES

A. Safety

1. For complete procedures, the Laboratory Safety Manual should be consulted.
2. Lab coats will be worn whenever analyzing drug evidence. Disposable gloves and safety glasses are provided for use but are optional.
3. Other safety equipment will be worn at the analyst's discretion. Visitors will be provided safety equipment as needed.
4. Procedures involving volatile chemicals (such as extractions) are to be performed in the fume hood.
5. Material Safety Data Sheets (MSDS) should be consulted when using a new or unfamiliar chemical. Those specific to the narcotics operation are located in the hallway just outside the narcotics analysis laboratory.
6. No food or drink is allowed in the Narcotics Analysis room.
7. Analysts must wash hands thoroughly before leaving the laboratory if s/he handled drug substances, in accord with universal precautions (see Laboratory Safety Manual).

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8. If there is a conflict between worker safety and other mandates in this SOP, worker safety considerations take precedence over other procedures (e.g. a noxious exhibit with packaging may be worked on in the hood if the analyst's safety would otherwise be compromised.)

B. General Procedures

Submitted compounds may take the form of powder, solid amorphous or crystalline material, liquid, resin, paper, pharmaceutical tablet or capsule, or plant material. A visual examination of the physical appearance of the submitted material will aid in reducing the scope of the analysis and should be the first step in the examination of any suspected controlled substance.

1. Screening tests

Screening tests (which can include presumptive tests) aid an analyst in narrowing the scope of possibilities such that appropriate confirmation technique may be employed. Screening tests that are routinely employed are physical appearance, odor, some color tests, microscopic examination and suggested possibilities the officer indicates on the envelope.

2. Presumptive tests

A presumptive test requires that an analytical test be performed, the result of which eliminates some drugs from consideration. These tests also indicate which class of controlled substance *might be* present, though it does not identify which drug *is* present. Presumptive tests include color tests and microscopic examination in the case of marijuana.

Although there are exceptions, the great majority of all controlled substances can be screened for by employing the following color tests: cobalt thiocyanate (C1); cobalt thiocyanate followed by acidified stannous chloride (C1a); Marquis (C2); Mecke (C3); Froehde (C4); p-DMBA also known as Van Urk's (C5); Dille-Koppanyi (C6); Duquenois-Levine (C7) and Wagner's (C8). The actual color test(s) selected varies depending on the physical appearance of the submitted material.

3. Confirmatory tests

Confirmatory tests are analytical tests with a higher degree of discriminating power than a presumptive test. These tests serve to identify the controlled substance in the sample. Based on how discriminating the analytical

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technique is, one confirmatory test may be used or a few tandem tests may be required (see guidelines below).

4. Confirmation criteria

The identity of a controlled substance is confirmed based upon the following criteria:

Category A	Category B	Category C
Mass Spectrometry	Microcrystalline tests	Color Tests
IR Spectroscopy	Gas Chromatography	
NMR Spectroscopy	Stereomicroscopy (Marijuana only)	
	Pharmaceutical Markings	

- a. When a category A technique is used, then at least one other technique from Category A, B, or C must be used. All Category A techniques must have data that are reviewable.
- b. When a Category A technique is not used then at least three different methods must be employed. Two of the three must be uncorrelated techniques from Category B. Microcrystal tests utilizing different reagents will be considered uncorrelated techniques.
- c. For Marijuana only: The specificity of the Duquenois-Levine reaction that causes a color change and subsequent extraction of the color is high enough that in combination with stereomicroscopy used to identify specific morphological characteristics (see section E 1 below), these two tests serve as confirmation of marijuana.
- d. The Mass Spectra reference library is used to assist the analyst in the confirmation of controlled substances by GC/MS. The library cannot be used to distinguish optical isomers.
- e. When testing indicates that an evidence sample may contain a specific enantiomer controlled under the California Uniformed Controlled Substances Act , and an identification as such is required, the sample will be submitted to the DEA Western Region Laboratory in San Francisco to determine its exact optical activity.

C. Sampling

1. If the appearance of evidence or its packaging suggests that more than one type of drug could be present, each different type of drug should be analyzed.
2. If the samples within a group are similar in appearance, only one needs to be analyzed.
3. Specifically regarding samples packaged with an opaque material such as colored balloons: if the size and shape are similar among all of the items, at

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least one of each color or 10% of the total should be opened, whichever is greater.

4. If the samples within a group differ in appearance, then one item from each different group will be examined. At the analyst's discretion, after confirming the identity of the sample from the first population, if the presumptive test result for the second population is the same as the first, then the second population can be reported as consistent with the first population based on the presumptive test only.
5. When possible, an effort will be made to leave sufficient sample to permit re-analysis by an independent laboratory should such a request occur.
6. When all else is equal, it is generally best to analyze the largest sample. If the samples are equal in size, a randomly-selected item is used for analysis.

D. Weighing

1. At the analyst's discretion, either a net weight or a gross weight can be reported, except for items where there is minimal sample present. If the analysis is performed in these cases, the report will state "residue" and the identity; gross weights should not be reported.
2. Often the net vs. gross decision is influenced by the nature of the evidence submitted. For instance, evidence consisting of numerous individually plastic-wrapped items of similar appearance lends itself to be reported as a gross weight.
3. Resinous samples or samples that easily charge with static electricity will be impossible to separate from the packaging and thus must be reported with a gross weight.
4. For evidence submitted in numerous packaging units, alike in composition (e.g. ziplocks with hearts printed on one side), a net weight can be reported by obtaining a gross weight, weighing one of the packages and subtracting the appropriate weight for all of the packages. In this instance, "avg" will be appended to the reported net weight to indicate that it was derived from a subtraction method, e.g. 12.45 g net (avg).
5. Items that come in dosage units (e.g. tablets, paper squares, capsules, cigarettes) are generally not weighed, rather, the total number or estimated total number is reported.

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6. Occasionally sentencing enhancements necessitate a net weight to be obtained. Reasonable requests to reweigh samples originally reported with a gross weight will be honored.

E. Guidelines for Analysis of Controlled Substances

An abbreviated version of the routine examinations performed for each class of controlled substance analyzed in this laboratory appears in this section. Comprehensive analysis techniques as well as explanations of the terminology used in these descriptions can be found in the training manuals, section 6

The following procedures are not all-inclusive. The chemists have the training and expertise necessary to be allowed to implement and use acceptable chemistry practices (such as extractions, mass spectrometry data interpretation) not delineated here. Additionally, it must be noted that these procedures describe the minimum number and types of tests that must be performed. The analyst may always conduct more tests than what is described here, but not fewer.

1. Marijuana

A large portion of the cases submitted to the laboratory contain marijuana. Exhibits can include: leaves, seeds, flowers, stems, wet or dry plants, compressed plant material, cigarettes or resin.

- a. Botanical screen

Cystolithic Hair: The shape of the protruding cystolithic hair is significant: it is curved and tapered, reminiscent of a bear claw. Secondly, the presence of a calcium carbonate formation at the base of the hair is important.

Simple (or clothing) hair: Present on the underside of the leaf. The shape of the hair is not as characteristic as cystoliths: they are more numerous and longer but do not curve or taper in a well-defined way.

Confirming the presence of both types of hair on a sample of the plant material under a stereomicroscope constitutes a positive presumptive screen for marijuana.

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Alternatively, if a whole leaf is submitted, the characteristic shape of the leaf can be used as a presumptive screen (see marijuana training manual for more details).

b. Color Test

Duquenois-Levine

- 1) About 10 seconds after adding the Duquenois reagent and concentrated hydrochloric acid to the plant material, a violet color develops. The color deepens with time.
- 2) A fainter violet color extracts into the added chloroform layer from the Duquenois/acid solution.
- 3) Confirmation is achieved only if the botanical screen and color test are both positive.

c. Concentrated Marijuana (Hashish)

- 1) The Duquenois-Levine test is used as the presumptive test.
- 2) It is up to the analyst to select which of the following two confirmations to employ:
 - a. A sample of the material is smeared onto a glass slide. Cystolithic hairs are sought out via microscope. Upon addition of dilute HCl, effervescence verifies the cystolith's identity. If no hairs are detected, confirmation must come from GC-MS (see section b).
 - b. The resin or plant material is extracted with Petroleum Ether. A sample of this extract is introduced into the GC-MS. If the fragmentation pattern shows the presence of THC, this constitutes confirmation of concentrated marijuana.

d. Protocol for Marijuana Plant Submissions

- 1) Marijuana plants and other wet marijuana material need to be inspected and, if necessary, dried by an analyst.
- 2) If the material requires drying, the case is going to trial, or there is an imminent destruction scheduled, then the following procedure should be followed:

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- (a) Place the material in the drying oven along with appropriate laboratory number descriptor. If material is too voluminous for the drying oven, it may be air-dried.
- (b) Material consisting of the entire plant may have the leaves stripped away from the stems and roots. The stems and roots may be discarded.
- (c) Repackage the evidence. The Narcotics Record form should reflect that the material has been dried; if the material was photographed and stripped this should also be indicated.
- (d) It is optional to photograph the material. If they are taken, photographs should include the material and the associated evidence envelope.

2. Cocaine

Cocaine is the most common controlled substance encountered in the San Francisco Police Department Criminalistics Laboratory. Cocaine exists in the coca plant in the form of a base. The extraction techniques used at this time converts it to a salt. In the laboratory, it is encountered in both forms: the salt “powdered” form and the base “rock” form. The analytical procedure is approached in such manner as to distinguish between these forms.

a. Presumptive color test.

Cobalt thiocyanate is the screening reagent used to determine the presence of cocaine. A blue precipitate is immediately formed in

the presence of cocaine salt, while cocaine base is unreactive. Acidified stannous chloride is added which yields the characteristic blue color when cocaine base is present.

b. Confirmatory Microcrystalline Tests

Two crystal tests are performed using the gold chloride (hydrogen tetrachloroaurate) and platonic chloride reagents. The resulting crystals are observed under a microscope under polarized light at 100x. Typical shapes of these crystals are shown in the cocaine training module—section 6—as well as in the crystal codes Appendix of the SOP.

c. GC-MS

A portion of the sample is dissolved in slightly acidic water. Upon addition of an aqueous base, a cloudy precipitate forms. The

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analyte is extracted into an appropriate organic solvent which is then injected into the GC-MS.

3. Phenethylamines

In this class, phenethylamine refers to the backbone structure, derivatives of which include: amphetamine, ephedrine and methamphetamine.

a. Presumptive color test.

Marquis is the screening reagent used to determine the presence of phenethylamines. Bright oranges ranging from red-orange to brown-orange are immediately formed in the presence phenethylamines.

b. Confirmatory Microcrystalline Tests

Two crystal tests are performed using the gold chloride (hydrogen tetrachloroaurate) and platinum chloride reagents. The resulting crystals are observed under a microscope under polarized light at 100x. Typical shapes of these crystals are shown in the phenethylamine training module—section 6—as well as in the crystal codes Appendix of the SOP.

c. GC-MS

A portion of the sample is dissolved in slightly acidic water. Upon addition of an aqueous base, a cloudy precipitate forms. The phenethylamine is extracted into an appropriate organic solvent and injected into the GC-MS.

4. MDA and Derivatives**a. Presumptive color test.**

Marquis reagent produces a dark reaction ranging from deep brown-black to blue-black for the MDA, MDE, MDMA and their analogs.

b. Confirmatory Microcrystalline Tests

Two crystal tests are performed. For each of MDA, MDE and MDMA gold chloride (hydrogen tetrachloroaurate) is used. For MDA and MDE, gold bromide (tetrabromoaurate) is used. For MDMA, it is platinum bromide. The resulting crystals are observed under a microscope under polarized light at 100x. Typical shapes of these crystals are shown in the MDA and derivatives training module—section 6—as well as in the crystal codes Appendix of the SOP.

c. GC-MS

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For other analogs, a GC-MS run is the only alternative. A portion of the sample is dissolved in slightly acidic water. Upon addition of an aqueous base, a cloudy precipitate forms. This solution is extracted with an appropriate organic solvent and injected into the GC-MS.

5. Hallucinogens

This class of controlled substances encompasses a large variety of compounds, the most common of which are listed below. Others are analyzed with the procedures described in section 3/E/9 where the most commonly used method is GC-MS.

a. Ketamine**1. Presumptive color test.**

Cobalt thiocyanate is the screening reagent used to determine the presence of nitrogenous bases. Upon the addition of the reagent, a blue precipitate is formed. The color is less intense than that obtained for cocaine salt. If acidified stannous chloride is added, the characteristic blue color fades.

2. Confirmatory Microcrystalline Tests

Platinum iodide and platinum bromide reagents are each added to suspected ketamine. The resulting crystals are observed under a microscope under polarized light at 100x. Typical

shapes of these crystals are shown in the Hallucinogens training module—section 6—as well as in the crystal codes Appendix of the SOP.

3. GC-MS

As an alternative to the crystal tests, GC-MS may be performed. A methylene chloride extract of a basified aqueous solution is injected and analyzed.

b. LSD

LSD is presented in varied forms including infusions added to perforated paper squares, liquids, small tablets known as “microdots”, gelatin panes and sugar cubes or (rarely) in a powder form. Dosages are extremely small as microgram levels can produce psychedelic effects.

1. Presumptive color test.

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Acidified ethanol added to a sample on pDMBA-soaked (para dimethylaminobenzaldehyde) chromatography paper gives a purple color in the presence of indoles, pyrroles and tryptophan. LSD contains an indole skeleton, thus giving rise to a positive purple color change.

2. Confirmatory test

Any of the infused samples is cut into small pieces and extracted under ultrasonication with dilute HCl. The decanted solution is basified and extracted with methylene chloride. GC-MS is used to confirm the presence of LSD. One should be aware of an isomer called LAMPA which is easily distinguishable from LSD by mass spectrometry.

c. Mescaline

Mescaline is derived from the peyote plant, the common form of which is referred to as "buttons". It is rarely seen in the SFPD Criminalistics Laboratory.

1. Presumptive color test.

The addition of marquis reagent turns an extract of the plant material orange.

2. Confirmatory test

A series of tests is needed in order to confirm the presence of mescaline. First the plant material must be dried and ground. Extraction of mescaline is accomplished by soaking the material in alcohol and ammonium hydroxide overnight. Next, evaporate the alcohol and treat the residue with 0.5N hydrochloric acid. Clean with chloroform and basify with sodium carbonate. Run TLC with TAEA system (Toluene:Acetone:Ethanol:Ammonia; 45:45:7:3). Run prep TLC with TAEA, extract silica gel with methanol and run GC-MS.

3. For more details, refer to the Bureau of Narcotics and Dangerous Drugs, US DOJ manual.

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d. PCP

1. Presumptive color test.

Cobalt thiocyanate is the screening reagent used to determine the presence of nitrogenous bases. Upon the addition of the reagent, a blue precipitate is formed. The color is less intense than that obtained for cocaine salt. If acidified stannous chloride is added, the characteristic blue color fades.

2. Confirmatory Microcrystalline Tests

Two tests are used to determine the presence of PCP. For each, dilute HCl is added to the unknown followed by Potassium Iodide or Hydrobromic acid. The resulting crystals are observed under a microscope under polarized light at 100x. Typical shapes of these crystals are shown in the Hallucinogens training module—section 6—as well as in the crystal codes Appendix of the SOP.

3. GC-MS

A methylene chloride extract from a basified water solution of the analyte can be run on the GC-MS and analyzed.

e. Psilocin and Psilocybin

Psilocybin and psilocin are constituents of over 40 species of mushrooms. The species fall into five genera, headed by Psilocybe and Stropharia. The other three are Panaeolus, Conocybe and Copelandia. The two most common mushrooms encountered are *psilocybe mexicana* and *stropharia cubensis*.

1. Screening test

A visual characterization can be a screening test. The most common psilocybin mushrooms have a distinctive odor and typically have bluish stains, primarily on the flesh of the stems.

Other varieties exist that do not exhibit this characteristic, therefore the only true screen is to perform the color test.

2. Presumptive color test.

The Weber test is used to indicate the presence of psilocin, not psilocybin. The reagent is freshly made from Fast Blue Salt B

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in water prior to performing the test. Upon adding this to the crushed plant material, a bright red color forms in the presence of psilocin. When concentrated HCl is added, the red turns blue.

3. Confirmatory test

One tenth to one quarter of a gram of dried mushroom is added to a large test tube. Methanol is used to extract psilocin under ultrasonication. The residue may be cleaned with petroleum ether, diluted in methanol and injected into the GC-MS. Upon coming into contact with the hot injection port, psilocybin converts to psilocin, therefore, coupled with the Weber test, the only component that can be confirmed as present is psilocin.

6. Opiates

Compounds derived from *Papaver Somniferum*, take two forms—natural and synthetic. The natural compounds are those alkaloids that are directly extracted from the opium poppy. Greater than 25 compounds have been identified, but the most common are morphine and codeine. Chemically synthesized products from opium precursors include heroin, oxycodone and hydrocodone.

a. Presumptive color tests

1. Marquis (C2) will form a purple color reaction in the presence of an opiate.
2. Mecke (C3) will form a green to blue color reaction depending on the opiate present. For example, codeine gives a green/blue, while heroin yields a dark green.
3. Froehde (C4) will produce a range of colors depending on the opiate present. For example, heroin produces a dark purple.

b. Confirmatory tests (GC-MS)

While crystal tests are known for members of the opiate family, GC-MS is the primary tool used to confirm the presence of a controlled substance. See the training manual for specifics on crystal tests as well as appendix containing the crystal codes and photos of crystal forms. Basified methylene chloride extractions are preferred for most opiate confirmations as some compounds in this class are not soluble in petroleum ether.

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c. Opium

The color and confirmatory tests described above are suitable for raw opium. Upon analysis by GC-MS, one will observe papverine and thebaine among other compounds. These confirm the opium mixture.

d. Methadone

While not strictly an opiate, it is included in this section as its association with heroin is undeniable since it is used as a tool for addicts to avoid serious withdrawal symptoms from heroin.

1. Methadone typically comes in one of two forms: tablets and a liquid suspension often pink and sweet-smelling.
2. Presumptive test
The addition of Marquis reagent yields a red color.
3. Confirmatory test by GC-MS
A basified methylene chloride extract is injected and analyzed.

7. Pharmaceuticals

Evidence items submitted are usually in the form of a tablet, capsule or liquid suspension of the drug. All pharmaceuticals containing small molecules

(molecular weight < 600 amu) can be analyzed in this laboratory. Proteins with high molecular weight may need to be sent out for analysis if confirmation is necessary.

Not all pharmaceutical exhibits require confirmation for the district attorney's office. If the charge is an 11350 H&S violation (possession), or the controlled substance is found to be a benzodiazepam by the manufacturer's pharmaceutical markings, a confirmation is not required for the district attorney's office before the preliminary hearing. If the charge is an 11351 or 11352 H&S violation (possession for sale or sale), the exhibit needs to be confirmed.

a. Primary Examination

Manufacturer's markings, the physical form of the pharmaceutical, its shape and color are all used to determine the identity of a submitted unknown. Several references exist that aid the analyst in identifying a pharmaceutical. Those references that are used in this laboratory include:

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1. Drug Identification Bible
2. Ident-a-drug
3. Med-scan
4. Physician's Desk Reference (PDR)

Additionally, if the references do not list the pharmaceutical, the California Poison Control Center may be employed for help by calling 1-800-523-2222.

Appropriate color tests can be selected based upon the markings. It should be noted that inert diluents are added to tablets to bring them to an appropriate size; the concentration of the drug can affect the strength and kinetics of the color test, thus it is important to be aware of low concentration formulae and possible false negatives due to the presence of small amounts of analyte.

b. Confirmatory tests

As stated in the introductory portion of the pharmaceuticals section, not all exhibits need to be confirmed. For those items that need confirmation, either crystal tests or GC-MS can be performed after appropriate preparation. More details on these methods can be found in the training manual.

8. GHB and Derivatives

Gamma Hydroxybutyric acid (GHB) cyclizes in an acidic environment to Gamma Butyrolactone (GBL). Thus, to confirm the presence of GHB, an approach that separates the two is necessary. Another common variant of these chemicals found in this laboratory is 1,4 butanediol; it is used in the same way as the so-called "date rape drug" since the body metabolizes it to form GHB. Each of these forms is illegal as a schedule II controlled substance or analog as defined by the Health and Safety code.

Often suspected GHB or its analogs are submitted as liquid solutions of the compound. Plastic water, soda, or sport drink bottles can be a clue that one should test for this family of drug. The following procedure is a suggested route to confirmation.

a. Presumptive tests

1. **Color test screen.** The presence of an alcohol can be analyzed by using the ceric ammonium nitrate test. A positive presumptive test indicates that 1,4 butanediol could be present rather than GHB or

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GBL. Two 1:6 diluted ceric ammonium nitrate solutions are made in test tubes. Next, 4-5 drops of the analyte is added to one test tube, the other remaining as a negative control. A change in color from light orange to red indicates the presence of an alcohol in the analyte.

2. **pH screen.** This optional test is useful for determining which of GHB or GBL is present. Pure GHB in water yields a neutral to slightly basic pH reading (~9), while GBL yields a slightly acidic (~4-5) reading. 1,4-butanediol also gives a slightly acidic reading, however, the color test would indicate that this compound may be present.
3. **GC-MS screen.** An aqueous sample of the unknown is diluted with either methylene chloride or methanol and injected into the GC-MS. The presence of GBL could be due to the presence of neat GBL or from a cyclized GHB; its detection indicates that work toward confirming which is present is warranted.

b. Confirmatory tests

1. GC-MS

This method shows how to confirm GHB.

Drying to a solid:

- 1) Evaporate 1-2 mL of liquid sample to obtain a solid. Experience shows that the more viscous the liquid, the more concentrated the unknown.
- 2) Remove a small portion of the solid material and place in a GC-MS vial.
- 3) Add 5 drops of BSTFA (with 1% TMCS), then approximately 2mL ethyl acetate, cap the vial, shake to mix and heat at 70°C for 5-10minutes.
- 4) Dilute a portion of this mixture 1:4 in ethyl acetate.
- 5) Run GC-MS.
 - aa. GBL main peaks at $m/z = 42, 86, 56$
 - ab. GHB + BSTFA peaks at $m/z = 147, 73, 45, 117, 233, 59, 133.$

2. FTIR

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- a) Once a sample is dried to a solid as in 1) above, it can be mixed with KBr and introduced to the diffuse reflectance assembly. Standards have been run and added to the spectral library, thus the presence of GHB should be readily apparent.
- b) Alternatively, since GBL is a liquid at room temperature, a sample presumed to be GBL can be added to a salt plate and run in transmission mode. Care should be given that the sample is dried to a viscous liquid that does not contain water as moisture can damage the salt plates. GBL is also in the spectral library.

3. Crystal Tests

See the Crystal Forms Appendix of the SOP for details and photos of the expected crystal form for GHB.

9. Barbiturates

Barbiturates were commonly prescribed sedatives before physicians began to replace them with benzodiazepines. They are rarely seen in the laboratory. However, the majority of them remain controlled substances.

a. Presumptive color test (Dille-Koppanyi)

The Dille-Koppanyi color test is a two-step process. The addition of a solution of cobalt acetate to the analyte is followed by 5% isopropylamine. The reagent will turn violet in the presence of a barbiturate.

b. Confirmatory tests

1) GC-MS

Barbiturates are unusual controlled substances in that the acidified form is the neutral form. Thus, acidic extractions must be performed. The solvent is then run by GC-MS to determine what compound is present.

2) Crystal tests

Some crystal tests exist for the barbiturate class. The narcotic analysis laboratory's Microcrystal Test reference notebook should be consulted for renditions of the expected

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forms. Additionally, the crystal forms appendix at the end of this SOP may contain some useful information.

10. Negative

Using the scientific method of hypothesis, test, conclude, an analyst first forms an initial opinion of what a submitted exhibit could be based on experience and training. A presumptive test is performed. If it fails to lend credibility to the hypothesis of what type of drug the exhibit is, a new hypothesis is formed and another test is conducted. An unknown that fails to give expected results for a series of presumptive tests can be deemed negative for the presence of common controlled substances.

- a. For the purposes of defining a presumptive test reaction, a significant color change is defined as one whose kinetics cause the reaction to occur in less

than one minute after the addition of the reagent and that cause a bright primary color to form.

- b. In this laboratory, the prescribed series of presumptive tests to show that no common controlled substances are present is the following:

(C1a) Cobalt thiocyanate/acidified stannous chloride—no blue color.

(C2) Marquis—no significant color change.

(C3) Mecke—no significant color change.

(C4) Froehde—no significant color change.

(C8) Wagner—no shiny, floating precipitate.

OPTIONAL: if submitted form suggests LSD, (C5) DMBA—no purple observed.

- c. A conclusion of NCCS is made.
- d. Plant material need only fail the botanical screen and (C7) Modified Duquenois-Levine or Weber tests to conclude “negative” for the presence of a controlled substance.

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- e. It remains at the analyst's discretion to use more than the minimum methods prescribed here.
- f. A material that is confirmed through markings, GC-MS or other means to contain uncontrolled material can either be reported as negative or not controlled, whichever is most appropriate.

11. Unclassified

Unknowns that do not fall neatly into any of the categories above need to be analyzed as follows:

Plant material. Unknown plant material that is not marijuana can be analyzed by stereomicroscopy. If no feature indicates a familiar controlled substance but it is still suspicious, two extractions can be performed. A sample of the material is placed in a test tube and either petroleum ether or methanol is added. The extract is filtered through a cotton-plugged pipette and run by GC-MS.

Unknown powder or unknown pill. The typical approach to these samples that have failed presumptive testing is to place methanol into a GC-MS injection vial, adding a small sample of the unknown and running GC-MS on the extract. If a controlled substance is found, the standard should be run to obtain the GC retention time and confirmation is achieved. Note that if a barbiturate is a possibility, an acidic extraction must be run. Other options are to perform a basic extraction and run GC-MS, use FTIR, UV-Vis or crystal tests if the evidence warrants such measures.

F. Work Progression of a Narcotics Case

- 1. During Analysis
 - a. Before a case is opened, the envelope is inspected and the integrity of the seal is verified. (Refer to section 2.B.3.c for definition)
 - b. The envelope is initialed and dated. If additional sealing was performed (e.g. tape and initials were added), this is indicated on the record form (see d. below).
 - c. The envelope is cut open, usually across the back of the top flap.
 - d. A Narcotics Record Form (currently version 1.6) is filled out with the analyst's name, the laboratory number (LN) and the SFPD incident

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number, date received from courier, condition of seal and any comments.

- e. The contents of the envelope are removed and the item count and description is matched to the summary on the exterior of the envelope. Discrepancies are noted on the Narcotics Record Form.
 - f. If not already done, the interior packaging is labeled with the unique case identifier. Additionally, the analyst initials the packaging.
 - g. Exhibit numbers supplied by the officer are used. If none exist, the analyst may assign one by writing it on the evidence item or packaging, as well as on the Narcotics Record Form. Alternatively, exhibit descriptors can self-identify items (i.e. 22 rocks, 37 tablets, etc.)
 - h. The analyst weighs the material and indicates if it is a net weight (no packaging included) or gross weight (packaging included).
 - i. As the analysis is in progress, the tests used for a given exhibit are recorded on the Narcotics Record Form. Similarly, the observations made are also recorded. A list of the most commonly used codes appears on the record form. A more comprehensive list of tests used is in Appendix B.
 - j. If GC-MS, FTIR or other reviewable data are generated, the spectra are printed out and initialed by the analyst. If multiple pages are present, a "1/3" type designator must also be present. If the case identifier is not on the printout, it will be added by the analyst.
 - k. If multiple evidence items are present, the next one is analyzed similarly. When all of the items are analyzed, the form is signed and dated by the analyst.
 - l. The contents of the envelope are repackaged as close as possible to how it was originally submitted. Any items introduced by the analyst (plastic bags, paper bindles) are to be clearly marked as added by the crime laboratory, for instance with "lab bag", "paper added by crime lab" or equivalent wording.
 - m. The envelope is staple-sealed. Only after this step may another case be opened.
2. Awaiting Review
 - a. The results of the examination are entered into the CABLE computer system.
 - b. The City and County of San Francisco/Crime Laboratory Original Record logo is embossed onto the Narcotics Record Forms over the analyst's signature. These forms are then placed in a box labeled "Narcotics Records for Review" in the hallway. If it is a QA day,

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these records are kept separate with a clip or folder so that the reviewer knows to initial the QA commitment statement and record these cases separately.

- c. Evidence envelopes will be stored in accordance with 6.1.1.2 D in a staple-sealed condition.
3. After Review
 - a. The Narcotics Record Forms are returned to the analyst with the date and initials of the reviewer entered in the appropriate section.
 - b. A photocopy of the Narcotics Record Form is made. Any supporting spectra are initialed and page-numbered by the analyst and stapled to this photocopy. These are placed in the "Completed Lab File Copy (Narcotics Record)" box in the hallway. The photocopied records are filed in numerical order in the hallway cabinet.
 - c. The original record form is placed back into the evidence envelope. It is sealed with evidence tape, stapled and initialed. Bulky items may be sealed with clear packing tape placed over the analyst's initials.
 - d. Sealed cases are placed in the box for return to the Hall of Justice.

4. Quality Assurance Program

A. Controls and Standards

1. Chemical Reference Standards: Authentic standards are maintained for many commonly encountered chemical compounds. A list of these reference standards is maintained in the narcotics laboratory.
2. Upon receipt by the laboratory of materials that are used as standards, the compounds are verified by GC-MS (or other suitable category A method) and the spectra are kept in the Primary Standards logbook, initialed by the person who verified the identity of the substance. If numerous packages from the same lot are purchased, only one need be confirmed with a Category A method. The package that was verified is marked with the analyst's initials, the date and "GC-MS OK" or "IR OK" based on the confirmation method used.
3. Microcrystalline Standards: A binder is maintained in the narcotics laboratory with pictures of the laboratory approved crystal forms necessary for the

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identification of a drug by microcrystalline testing. Additionally, this appears as a separate appendix to this SOP for reference.

4. Commercially available compounds of reagent grade or higher (for instance solvents, acids or gases) do not need to be tested before use.
5. A negative control is run before every evidence item analyzed by GC/MS. The negative control solvent shall be the same solvent used for the evidence item.
 - a. A negative control (blank) shall be free of contaminants of controlled substances to be identified. If a controlled substance is detected in the negative control run, corrective measure(s) shall be implemented to ensure the pending evidence analysis is free from this contamination.
 - b. Corrective measures may include, but are not limited to, the following procedure(s):
 - i. Re-run the negative control
 - ii. Replace the negative control solvent
 - iii. Bake the column at a high temperature to elute contaminant(s)
 - iv. Change the septum
 - v. Change the column lining
 - vi. Consult service representative as necessary by instrument performance
 - c. The negative control is repeated until the result is free from contamination of any controlled substances of interest, and before evidence samples are run.
 - d. Corrective measures iv, v, and vi are recorded in the instrument log.

B. Reagent Control

1. All reagents utilized in analyses are prepared according to the guidelines set forth in Appendix A. Recipes are included in the Reagent Logbook.
2. The reagent will be labeled with the reagent name, the initials of the analyst who prepared it, and the date of preparation.
3. All reagent preparations will be recorded in the reagent log book.
4. Reagents will be verified on standards before use on case samples. The specific analyte used for verification is specified in Appendix A. If desired by the analyst, a previously confirmed sample of the analyte may be used to verify the reagent.

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5. The Narcotics Supervisor will check the efficacy of the reagents at each workstation with the frequency reported in the monthly QA checklist and discard and document any reagent that does not perform adequately. Alternatively, a lot may be prepared and checked; each workbench then receives a fresh replacement.
6. If the analyst notices that the response of a reagent is deficient before the end of the expiration period or that its composition has changed (color or precipitants), the reagent will be discarded and replaced immediately.

C. Quality Control Measures

1. General considerations

- a. Only one case will be open at one time.
- b. New butcher paper will be placed at each workbench daily, or sooner if needed.
- c. New butcher paper will be placed in common laboratory spaces monthly, or sooner if needed.
- d. Packaging for a case remains in the analyst's workspace, though samples from an exhibit may go to common areas of the laboratory for preparation and analysis.
- e. For Category A methods of analysis, libraries will be referenced to verify the identity of the unknown. If no match is found, bound references may be employed. If necessary, a standard will be run if no reference spectrum exists.

2. Monthly QA Responsibilities

By the 10th of each month, the following procedures will be carried out by the Narcotics Supervisor or his delegate and recorded in the "monthly QA check" notebook. The monthly QA reports and supporting spectra will be kept at least one year.

- a. The laboratory butcher paper lining in the bed of the fume hoods will be changed. It will be replaced sooner if necessary.

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- b. Check of the balances for accuracy will be conducted. Any two weights from among the available 10mg, 50mg, 1g, 10g, and 50g standards will be used and recorded on the QA check form. The weighed value must agree to within $\pm 10\%$ or $\pm 0.01\text{g}$ (whichever is greater) of the indicated value.
- c. Verification that technical reviews exceed 10% of all cases will be made. This includes all cases generating GC-MS spectra, and QC day cases. The supervisor will insure that it exceeds 1/10 of the number of cases reported in CABLE for the previous month. The number of reanalyzed cases is also recorded.
- d. With the frequency indicated on the monthly QA check form, reagents at each workbench will be checked for efficacy. Prescribed standards indicated on the form will be used to check the reagents. Any reagent that fails will be discarded and replaced. Notation will be made in the "Monthly QA check" notebook.
- e. A polystyrene standard will be run in the IR instrument and the spectrum kept in the instrument Calibration and Maintenance binder.
- f. A GC standard will be run on each GS-MS instrument and verification that the retention time is acceptable will be indicated on the form.
- g. A backup of computer data will be conducted. At a minimum, this will include backing up GC-MS TIC and spectral data from each GC-MS computer on the hard drive of the other. If numerous FTIR spectra or photos of microcrystals were collected in a given month, it is advisable to backup this data onto appropriate media as well.

D. Notes and Reports

1. All notes must be made on the Narcotics Record Form version 1.6. This includes the weight of the controlled substance, any tests performed, the observations made, the conclusions of the analyst and any other notes that would be necessary to recreate the analysis (i.e. H₂O wash, weights of bags for net weight reconstruction).
2. Spectra for category A methods must be appended to the form with the appropriate box checked indicating accompanying data.
3. All results are entered into the CABLE system and administratively reviewed. Upon completion of the review, the reviewer initials and dates the Record Form.
4. If the analysis occurred on a QA day, the box on the Narcotics Record Form should be checked indicating this fact; the reviewer (either administrative for

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appended data, or the technical reviewer at the time of the analysis) needs to initial by the box that the technical review has occurred.

5. Photocopies of the original report are filed in numerical order by lab number. These records are kept in-house on a space-available basis. Once the bay of file space in the laboratory is exceeded, records are boxed and sent to a recordkeeping warehouse. Departmental policy states that these records are to be kept at least 5 years.

E. Case Review

1. Administrative Review

- a. All cases are administratively reviewed.
- b. An administrative review includes:
 - 1) Insuring that the results of the analyses performed are consistent with the identity of the analyte reported (e.g. color tests results, crystal form).
 - 2) The type of test performed must also be appropriate. For example, insuring that the analyst did not indicate “pharmaceutical markings” and conclude “heroin”.
 - 3) A check of the CABLE entry to insure that the analyst’s report of the identity and weight of the material on the Narcotics Record Form is the same as what is in the computer.
 - 4) Insuring that all case identifiers recorded in CABLE are properly reflected on the Narcotics Record Form.
 - 5) Inspection of the Narcotics Record Form checking that the declaration is filled and that the form is signed and dated.
- c. In the event of an omission or discrepancy, the analyst will be requested to correct or supply data for the entry.
- d. After fixing an incorrect CABLE entry, the appropriate person (usually narcotics rebooking) will be contacted with the correction.

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- e. Upon completion of the review, the reviewer initials and dates the Record Form.

2. Technical Review

- a. Ten percent of cases are technically reviewed.
- b. The ten percent number is achieved in two ways. First, every case that requires instrumental data is technically reviewed. Second, one day per week (currently Wednesday), every case that is confirmed by microcrystalline tests is also technically reviewed either by generating a GC-MS spectrum, concurrent agreement by a second analyst of the crystal form, photographing the crystal for future review or reanalysis.
- c. The technical review includes:
 - 1) Insuring that the analyses performed support the conclusion (color tests results, spectra, etc.)
 - 2) A check of the reviewable data insuring that it is consistent with the reported substance.
 - a) Reviewable data may include printed spectra or chromatograms.
 - b) For microcrystalline testing, contemporaneous documented peer review or photographs of the crystals serve as reviewable data.
- d. In the event of a discrepancy, the analysis in question must be verified and corrective action taken as per the procedures outlined in the QA manual.
- e. Upon completion of the review, the reviewer initials and dates the Record Form. For a QA day with instrumental data, the technical reviewer also initials by the "laboratory's commitment to quality assurance" checkbox. For a QA day involving contemporaneous review of crystal form, the technical reviewer initials the crystal form reported by the analyst.

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- f. A reanalysis of an analyst's case is not considered a strict technical review of the work, however, as with technical reviews, in the event of a discrepancy, the analysis in question must be verified and corrective action taken as per procedures in the QA manual. After the reanalysis is completed and results recorded on the reanalysis record form, the re-analyst initials and checks "ID OK" once it is confirmed that both analysts came to the same conclusion. Two photocopies are made of the reanalysis record form. The original is sealed in the evidence envelope; one photocopy is saved for compilation of monthly QA reanalysis numbers and the second photocopy is filed with the original analyst's report.

F. Competency Testing

1. At the end of each unit/ training module, an unknown sample or set of samples is given for testing. Upon satisfactory completion of the module, a memo will be written to the analyst to be filed in his or her Professional Development Folder. If any of the unknowns is incorrectly identified,

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remedial action must be taken. Please see the laboratory quality manual for more specific information on remedial actions.

2. Upon completion of the Narcotics Unit training program, a thorough set of unknowns must be correctly identified. The Criminalist will not be qualified for casework until this final competency test has been passed.
3. Upon completion of this test, a memo will be written stating that satisfactory completion has been achieved and a copy placed in the analyst's Professional Development Folder.
4. Upon qualifying for casework, the Criminalist must have a full review of all results for one month. The results will be co-signed by an experienced Criminalist who is qualified to perform narcotics casework. These cases should include a representative sample of the casework performed by the laboratory.

G. Proficiency Testing

Each Criminalist qualified to perform Narcotics casework must pass either an in-house or CTS proficiency test annually. In the event that the annual proficiency test is failed, remedial actions must be taken as stated in the laboratory's quality manual.

H. Courtroom Testimony Monitoring

Each Criminalist who provides expert testimony in narcotics must have his or her testimony monitored annually by direct observation, a review of the court records, or a survey to be completed by an attorney. For more details, please see the laboratory quality manual.

I. Laboratory Equipment

1. Gas Chromatograph-Mass Spectrometer (GC-MS)
 - a. The Narcotics laboratory is equipped with two Agilent Technologies 6890 Series Gas Chromatographs with Mass Selective Detectors (MSD). These are capable of producing Electron Impact (EI) spectra with unit resolution.

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- b. Calibration: The mass spectrometers are calibrated and tuned using the AutoTune function in the instrument software. The printouts generated by the AutoTune are placed in the Calibration Binder for that particular instrument. For more detailed description of the tuning and mass calibration of the mass spectrometers, please reference Appendix G.
- c. Maintenance: We have a maintenance contract with the manufacturer for all non-routine repairs. Laboratory personnel may perform routine maintenance jobs. All repairs and replacement of consumables must be recorded in the laboratory notebook for that particular instrument.

2. Fourier-Transfer InfraRed Spectrometer (FTIR)

- a. The Narcotics laboratory is equipped with one Thermo-Nicolet Avatar 360 FT-IR spectrometer.
- b. Calibration: The laser is used as an internal calibrator in FT-IR instruments, because it emits light at a known and constant frequency. All spectra are collected at precise laser-calibrated points, which generate wavenumber accuracy of $\pm 0.01 \text{ cm}^{-1}$. Additionally, a polystyrene standard is run monthly to insure that it is measuring absorbances correctly.
- c. If the spectrum generated after running the polystyrene standard is searched against the Hummel Polymer Sample Library, the atactic polystyrene spectrum should have a match exceeding 85. Additionally, three of the top five most intense peaks must contain $698 \pm 2 \text{ cm}^{-1}$, $1492 \pm 2 \text{ cm}^{-1}$, and $2924 \pm 2 \text{ cm}^{-1}$. If it fails to select the appropriate spectrum, have a sufficiently high match, the three peaks above are not the most intense or are out of range, another spectrum is collected. If this also fails, routine maintenance can be performed. If a subsequent spectrum does not agree, the instrument will be taken offline and the service representative called.
- d. Maintenance: The manufacturer performs all non-routine maintenance. Laboratory personnel may perform routine maintenance jobs. All repairs and maintenance must be recorded in the laboratory notebook for the instrument.

3. UltraViolet-Visible wavelength Spectrophotometer (UV-Vis)

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- a. The Narcotics laboratory is equipped with one HP model 8453 UV-Vis spectrophotometer.
 - b. Calibration: This instrument is only calibrated at the time of use.
 - i. A Holmium Oxide standard is run by placing the sample in the cuvette holder of the instrument.
 - ii. A spectrum is taken over the ~200-1100nm wavelength range.
 - iii. The peaks that must be observed to within ± 1 nm are as follows (listed in decreasing order of intensity):
 - 446 nm
 - 460 nm
 - 454 nm
 - 361 nm
 - 279 nm
 - 288 nm
 - iv. If the peaks are not present, out of the ± 1 nm window or have decreasing peak intensities in a different order than indicated in iii., another scan is performed and checked. For minor deviations of the wavelength calibration, the recalibration function in the ChemStation software can be performed to compensate. Please see the Reference manual for the ChemStation software for the exact procedures. If this also fails, routine maintenance can be performed. If a subsequent spectrum does not agree, the instrument will be taken offline and the service representative called.
 - c. Maintenance: The manufacturer or qualified service technician will perform all non-routine repairs. Laboratory personnel may perform any routine maintenance. All repairs must be recorded in the maintenance book for the UV-Vis Spectrophotometer.
4. Gas Chromatography-Flame Ionization Detector Spectrometer (GC-FID)
- a. The narcotics laboratory is equipped with one Agilent Technologies 6850 Series Gas Chromatograph with a Flame Ionization Detector.

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- b. Calibration: Since this instrument is used mainly for quantitative analyses, it shall be calibrated with a dilution series of the analyte of interest each time it is used.
- c. Maintenance: We have a maintenance contract with the manufacturer for all non-routine repairs. Laboratory personnel may perform and routine maintenance. All repairs must be recorded in the maintenance book for the GC-FID.

5. Microscopes

- a. The narcotics laboratory is equipped with four Nikon Eclipse E800 Polarizing Light Microscopes (PLM) and one Nikon SMZ800 Stereomicroscope.
- b. Maintenance: We have a maintenance contract with the manufacturer for repairs and annual maintenance procedures. Laboratory personnel may perform routine cleaning and alignments.

6. Analytical Balances

- a. The narcotics laboratory is equipped with two Mettler-Toledo B154 balances, one Mettler-Toledo PG802-S balance, one Mettler-Toledo PM300 Balance, one Mettler-Toledo AB104-S, and one Mettler-Toledo SB16001.
- b. Maintenance: We have a maintenance contract with the manufacturer for repairs and annual maintenance. Laboratory personnel may perform routine cleaning.
- c. Calibration: The manufacturer fully calibrates the balances annually. In addition, the Narcotics Supervisor performs a monthly calibration check of the balances.

7. Nuclear Magnetic Resonance Spectrometer (NMR)

- a. The Narcotics laboratory is equipped with one Anasazi model EFT 90 NMR Spectrometer. **This instrument is NOT in use for casework. Upon validation of procedures, implementation of this instrument will occur to allow this laboratory to use it for cases.**

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- b. Maintenance: The instrument is under warranty for five years (August 2003-July 2008). After this time, maintenance will be contracted through Anasazi. Routine magnet adjustments can be made by the analysts.
- c. Calibration: Performed on an as-used basis. A spectrum of tap water is acquired and the tuning is phased such that the resolution is optimized.

- i. A 5% ethyl methacrylate standard is used for shimming for H^1 data collection. A spectrum of the ethyl methacrylate is obtained. Peaks should be observed at:

- 6.1 ppm (peak of multiplet)
 - 5.5 ppm (peak of multiplet)
 - 4.3, 4.2, 4.1, 4.0 ppm quartet
 - 1.9 ppm (peak of multiplet)
 - 1.38, 1.30, 1.22 ppm triplet

Each at ± 0.1 ppm or $\pm 5\%$, whichever is greater.

- ii. The integral of the peak at 6.1 ppm should be set to 1.00, the others should integrate to 1.00, 2.00, 3.00 and 3.00 $\pm 10\%$ respectively.
 - iii. If either the peak position or integral fails the tests above, the analyst should first check and correct the position of TMS to 0.0 ppm. If this does not help, a shim should be conducted. Cleaning of the head, ensuring that the sample is spinning and fixing it if it is not, and only if necessary, a retuning of the magnet can all be conducted by the analyst.
 - iv. If further spectra collection still does not meet the criteria above, then the instrument is taken offline and a service representative contacted.

8. Computer Equipment

- a. The computers in the Narcotics Analysis laboratory are interfaced with instrumentation. A short inventory follows:
 - i. GC-FID

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- ii. 2 GC-MS
 - iii. FTIR
 - iv. Nikon microscope mounted camera
 - v. UV-Visible
- b. As a part of the monthly QA check conducted in the unit, data from each of the two GC-MS computers are backed up onto the hard drive of the other computer. This is to ensure that in the short-term, data will be retrievable in a copy other than the hardcopy printout supplied with the report.

5. Appendices

Please see the attached Appendices A-I which were referenced in the above text.

Appendix A.

Reagent Recipes

APPENDIX A. Reagent Recipes

Acidified Stannous Chloride—Dissolve 5 g stannous chloride in 10 mL concentrated hydrochloric acid and dilute with water to 100 mL.

Cobalt Thiocyanate—2% w/v cobalt thiocyanate in water

Davis Silver—1 g silver nitrate to 10 mL water and add 1.5mL ethylenediamine

Dille-Koppanyi—Consists of two reagents, added to the unknown in succession.

- Reagent 1: “Cobalt acetate” 0.1 g cobaltous acetate, tetrahydrate is added to 100mL absolute methanol. Then, 0.2mL glacial acetic acid is added.
- Reagent 2: “isopropylamine” 5mL of isopropylamine is added to 95mL absolute methanol.

Duquenois—Dissolve 50 drops acetaldehyde and 4 g of vanillin in 200 mL of ethanol.

Froehde—Dissolve 0.1 g molybdic acid or sodium molybdate in 20 mL of hot concentrated sulfuric acid

Gold Bromide—5% gold bromide (hydrogen tetrabromoaurate (III)) w/v in water

Gold Chloride—5% gold chloride (hydrogen tetrachloroaurate (III) trihydrate) w/v in water

Marmes—Dissolve 3 gm cadmium iodide in 18 mL water containing 6 g potassium iodide

Marquis—Add 8-10 drops of 40% formaldehyde solution to 10 mL concentrated sulfuric acid

Mercuric Chloride—5% w/v mercuric chloride in water

Mecke—Dissolve 0.25 g selenious acid in 25 mL concentrated sulfuric acid

p-DMBA—2% w/v p-dimethylaminobenzaldehyde in ethanol. Soak solution onto filter paper and allow to dry. Cut Filter paper into small test strips and store in tightly sealed light resistant jar.

Platinic Bromide—5% w/v platinic bromide in D.I. water

Platinum Chloride—5% w/v platinic chloride (hydrogen hexachloroplatinate(IV)) in water

Platinic Iodide—55% w/v platinic iodide in water

Potassium Iodide—5% w/v potassium iodide in water

Appendix A.

Reagent Recipes

Stannous Chloride—see Acidified Stannous Chloride

Wagenaar's—Add ethylenediamine to a 5% solution of copper sulfate until the solution becomes a dark violet in color

Wagner's—Dissolve 1.27 g iodine and 2.75 g potassium iodide in 5 mL water and dilute to 100 mL with water

Weber—0.01% w/v Fast Blue B in water. This preparation is not to be stored, rather it is prepared fresh before each use.

Appendix B.

Codes

APPENDIX B. Codes

Below are the codes that represent the most commonly used narcotic tests used by the S.F.P.D Crime Lab

Color Test terms

C=Color test

C1=Cobalt Thiocyanate

C1a=Cobalt Thiocyanate/acid

C2=Marquis

C3=Mecke

C4=Froehde

C5=DMBA/Van Urk

DMBA=p-Dimethylbenzaldehyde

C6=Dille-Koppanyi

C7=Duquenois-Levine

C8=Wagner's reagent when used to check for the presence of alkaloids

Crystal Test terms

X=Crystal test

X2=Gold Chloride or hydrogen tetrachloroaurate

X3=Platinic Chloride

X4=Potassium Iodide

X8=Wagner's when used for a crystal test reagent

X26=Platinum Bromide

Appendix B.

Codes

Other Test, Observation and Conclusion terms

M=Microscopic examination

M+=botanical features indicative of marijuana observed

Markings or mkg=Pharmaceutical manufactured markings

bl=blue

bk=black

br=brown

dk=dark

grn=green

lt=light

neg or (-)=negative (no significant color change or no botanical features)

=positive result

nr=no reaction,

org=orange

p=purple

pk=pink

v=violet

→=turns to

↓=extracts

g=grams

mL=milliliters

avg= average

NCCS=No Common Controlled Substances Detected

Appendix B.

Codes

Instrumentation terminology

CABLE=Computer Assisted Bay Area Law Enforcement

FT=Fourier Transform

GC-MS=Gas Chromatography-Mass Spectroscopy (technique) OR
Gas Chromatograph-Mass Spectrometer (instrument)

IR=Infrared light or infrared spectroscopy

NMR=Nuclear Magnetic Resonance Spectrometry

UV=Ultraviolet spectrophotometry

UV-Vis= Ultraviolet-Visible light spectrophotometry

Appendix C.

Narcotics Record Form

APPENDIX C. Narcotics Record Form

See the next page for a copy of the record form.

APPENDIX D. Narcotics Reanalysis Form

See the next page for a copy of the Narcotics Reanalysis form.

APPENDIX E. Proposition 115 Interview Form

SAN FRANCISCO POLICE DEPARTMENT CRIME LAB

CONTROLLED SUBSTANCES PROPOSITION 115 INTERVIEW FORM

Lab Number of Analyzed Evidence Envelope _____

BEFORE OPENING THE ENVELOPE, ASK THE FOLLOWING QUESTIONS OF THE CRIMINALIST AND CIRCLE THE ANSWER

Pre-interview question for teleconference only: Can you both hear and see me via this teleconference connection? Yes

- 1. What is your name and title? _____
- 2. Are you an employee of the San Francisco Police Department Crime Lab? Yes No
- 3. Do you regularly test for controlled substances as defined by the Health and Safety Code? Yes No
- 4. What education have you received that is relevant to your job duties?

5. What is your work experience in narcotics testing? _____

6. Do you recognize this envelope? Yes No How? _____

7. When you received this envelope, was it in a sealed condition? Yes No

NOW OPEN THE ANALYZED EVIDENCE ENVELOPE AND SHOW THE CONTENTS TO THE CRIMINALIST

8. Do you have a present recollection of the test results in this case? Yes No

(If the answer is "Yes" ask why s/he has a present recollection of testing the substance(s) and proceed to question 10) _____

8A. Do you routinely write a record of your test results? Yes No

8B. Do you personally write down the test results immediately after performing the tests? Yes No

8C. Where do you write down these test results? _____

8D. Do you recognize your writing on the RECORD form in this case? (show form) Yes No

8E. Is that writing a true statement of the test results? Yes No

8F. Did you personally test the evidence in this case? Yes No

9. Did you use your standard operating method to test this evidence? Yes No

10. How do you know you used your standard operating method? _____

11. Approximately how many times have you used this method to test this type of drug? _____
12. Are these testing methods accepted in the relevant scientific community? Yes No
13. Were both presumptive and confirmatory tests performed in this case? Yes No
14. How do you know both presumptive and confirmatory tests were performed? _____

15. Did the results appear to be both valid and unexceptional? Yes No

16. What did you record as a result of your tests?

	<u>Type of Drug</u>	<u>Weight</u>
Item #	_____	_____
Item #	_____	_____
Item #	_____	_____
Item #	_____	_____
Item #	_____	_____

RETURN ALL ITEMS TO THE ENVELOPE AND SEAL THE ENVELOPE.

Name of Interviewer _____

Date

Time

APPENDIX G. Instrument Tuning and Calibration Check for GC-MS

Instrument Tuning and Calibration Check for an Agilent Technologies 6890 Gas Chromatograph equipped with a Mass Selective Detector (MSD)

The MSD comes equipped with an AutoTune program, which checks and adjusts instrument parameters and calibrates the mass spectrometer. This program will tune and calibrate an instrument consistently, independent of the operator. Assembly of the results into the calibration binder provides a chronicle of system performance over time. Tuning and calibration utilizes perfluorotributylamine (PFTBA), which fragments over a wide range of masses, and is therefore deemed a suitable calibrant by the instrument manufacturer and is widely used.

Some criteria of an acceptable tune and calibration are as follows:

Masses at 69.0, 219.0, 502.0 \pm 0.2 Da

Peak width at 50% (pw50) \leq 0.6 Da (checked at m/z 69, 219, and 502)

Total number of peaks \leq 160

Relative abundance of m/z 502 $>$ 2%

Relative abundance of m/z 219 $>$ 30%

Isotopic ratio of m/z 69:70 between 1.0-1.3%

Isotopic ratio of m/z 502:503 between 9.4-11.5%

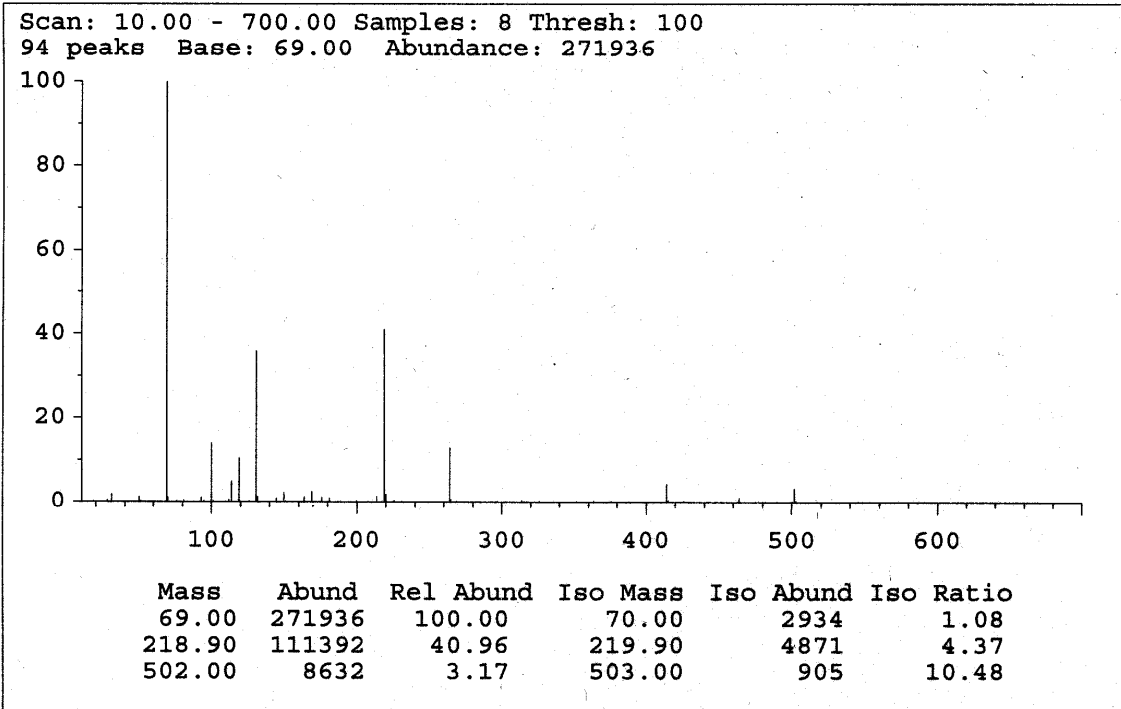
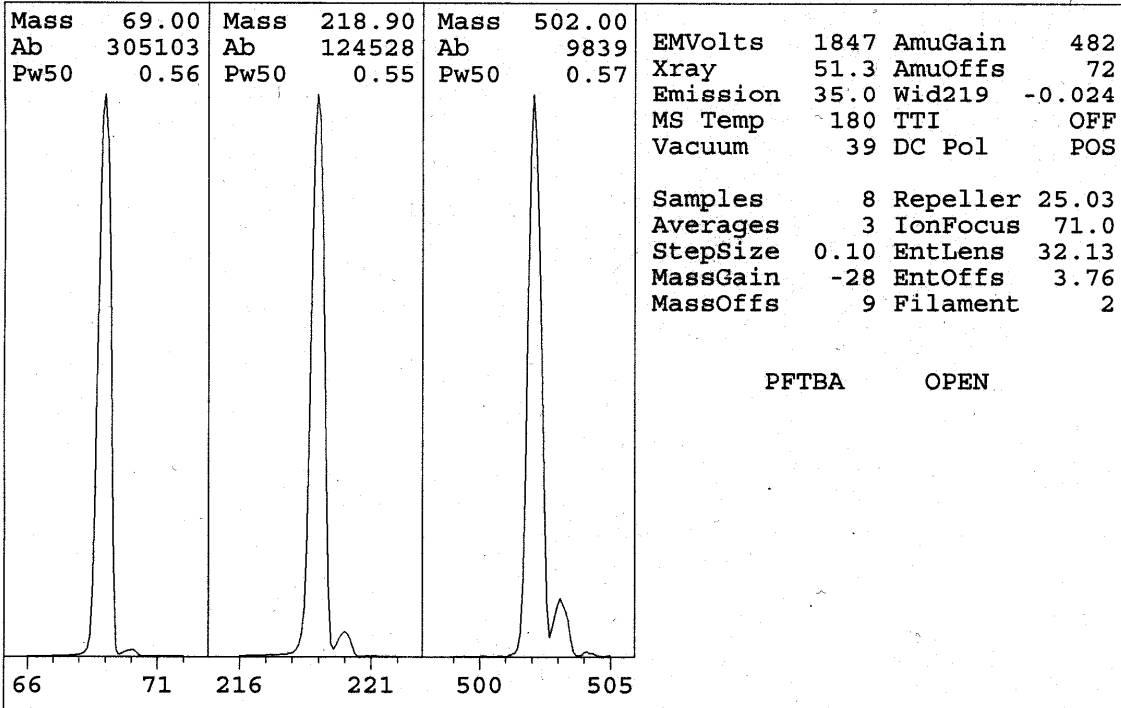
The AutoTune procedure will be performed on a weekly basis, and a printed record kept in a binder near the instrument. A qualified operator will initial this record. The instrument will also be tuned after any routine maintenance is performed on the detector.

If the autotune is outside these ranges, another autotune will be performed. If this also fails, routine maintenance can be performed. If the autotune still fails, the instrument will be taken offline and the service representative called.

An example of an acceptable autotune report is presented on the following page.

Example of An Acceptable Autotune Report

HP5972 Standard Spectra AutoTune
 Instrument: GC/MS #1
 Mon Jan 28 07:47:13 2002 C:\HPCHEM\1\5972\ATUNE.U



APPENDIX H. Monthly QA check Form

The following three pages outlines the procedure followed for the monthly QA check of the Narcotics section.

San Francisco Police Department
 Criminalistics Laboratory
 Narcotics Analysis Unit



Monthly QA Check

For the month of _____, 20__

To be conducted by the 10th of the following month

◆ The butcher paper lining in the bed of the fume hoods was changed. _____, _____
 Initial Date

◆ Calibration of the balances was checked. _____, _____
 Initial Date

Workbench	Weight 1 used _____	Weight 2 used _____
#1		
#2		
#3		
#4		

Balance	Weight 1 used _____	Weight 2 used _____
Mettler AB104S		

Balance	Weight 1 used _____	Weight 2 used _____
Mettler SB16001		

Were the results within $\pm 10\%$ or $\pm 0.01\text{g}$ (whichever is greater) of the indicated value?
 Yes No

If no, what corrective action was taken? _____

(continued)

Monthly QA Check

For the month of _____, 20____

- ◆ At least 10% of cases were technically reviewed.

Total number of CABLE entries: _____ 10% Goal:

The goal was achieved in the following ways:

- Number of cases with GC-MS or FTIR reviewable spectra: _____
- Number of cases analyzed and reviewed on QC days: _____
- Total:

- ◆ Number of reanalyses performed:

- ◆ Reagents at each workbench were checked for efficacy with the frequency indicated below (with initials by the check mark when completed). Alternatively, a common lot may have been checked and each workbench received a fresh replacement.

Reagent	Testing Standard	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
Marquis	Heroin	✓		✓		✓		✓		✓		✓	
Mecke	Heroin				✓						✓		
Froehde	Heroin					✓						✓	
Cobalt Thiocyanate	Cocaine Base + Acid		✓						✓				
Stannous Chloride (acidified)	Cocaine Base + Cobalt Thiocyanate		✓						✓				
H ₂ PtCl ₆	Cocaine Base		✓			✓			✓			✓	
HAuCl ₄	Cocaine Base			✓			✓			✓			✓
Platinic Bromide	MDMA	✓			✓			✓			✓		
Wagner's	Codeine		✓				✓				✓		
Gold Bromide	MDA			✓						✓			
Marme	Codeine				✓						✓		
Mercuric chloride	Methadone					✓						✓	
1:1 EtOH/HCl/DMBA	LSD						✓						✓
Platinic Iodide	Ketamine	✓						✓					
Potassium Iodide	Codeine												✓

Indicate any reagents that were replaced and why _____

 Initial Date

(continued)

Monthly QA Check

For the month of _____, 20__

◆ A polystyrene standard was run in the IR instrument. _____, _____
Initial Date

- Did the search match atactic polystyrene >85%? Yes No
- Were the frequencies $698 \pm 2 \text{cm}^{-1}$, $1492 \pm 2 \text{cm}^{-1}$, and $2924 \pm 2 \text{cm}^{-1}$ among the top five most intense absorbances? Yes No

If either were answered “no”, what corrective action was taken?

◆ GC standard run on each GC-MS instrument. _____, _____
Initial Date

- The retention time of peaks is within $\pm 10\%$ or ± 30 seconds (whichever is greater) of previous month’s value. Yes No

If answered “no”, is there a reasonable scientific explanation (e.g. column shortening)? Otherwise, what corrective action was taken?

A backup of computer data was performed. _____, _____
Initial Date

 Signature of person responsible for QA check

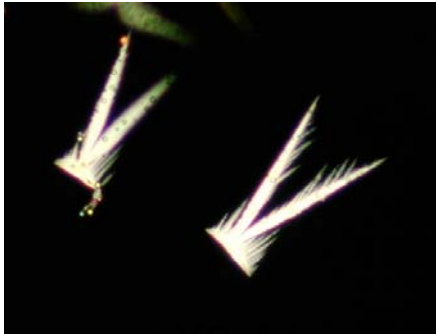
 Date completed

APPENDIX I. Approved Crystal Forms

Listed and shown below are the crystal forms that are approved for use in this laboratory; they are indicative of a particular analyte under specific conditions. Accompanying the photograph of the crystal forms are the reagents necessary to form the shown crystal. Please read further references for more specifics on crystal forms, reagent descriptions and experimental conditions (e.g. Clarke's "Isolation and Identification of Drugs").

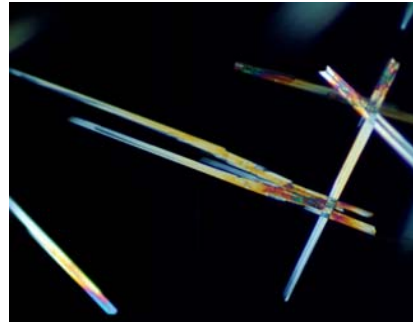
Appendix I.

Approved Crystal Forms



X2-A1

Methamphetamine/H3PO4/HAuCl4



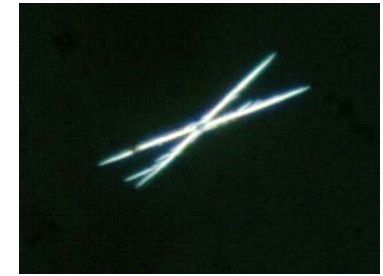
X2-A2

Methamphetamine/H3PO4/HAuCl4



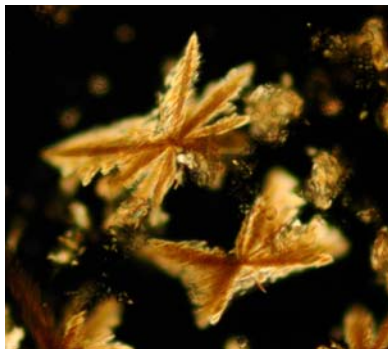
X2-A3

Methamphetamine/H3PO4/HAuCl4



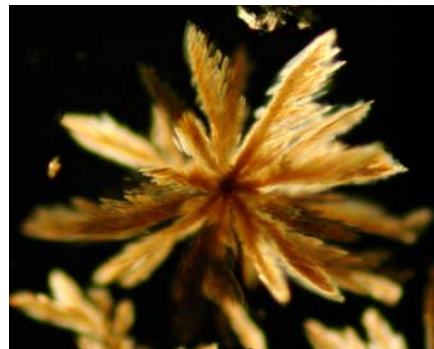
X2-B1

Cocaine/dil. HCl/HAuCl4



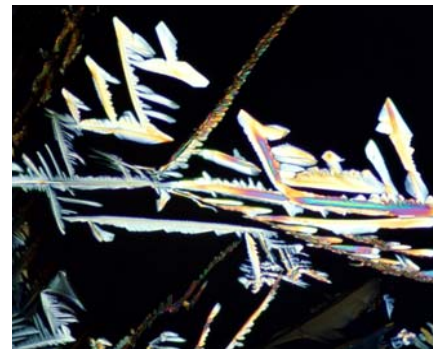
X2-C1

3,4-MDMA / dil. HCl /HAuCl4



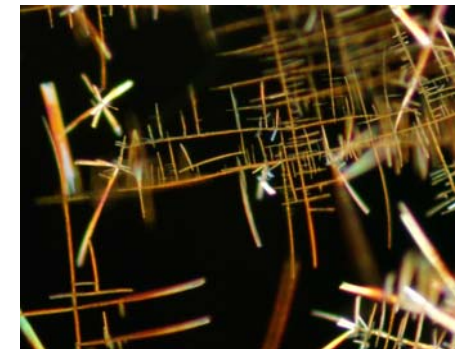
X2-C2

3,4-MDMA / dil. HCl /HAuCl4



X2-D1

3,4-MDA / dil. HCl /HAuCl4



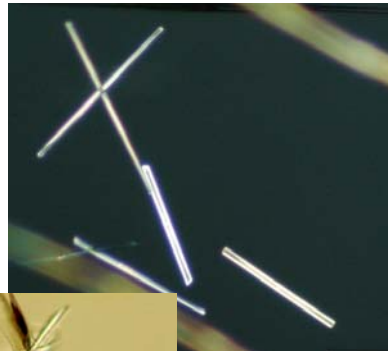
X2-E1

3,4-MDE / dil. HCl /HAuCl4



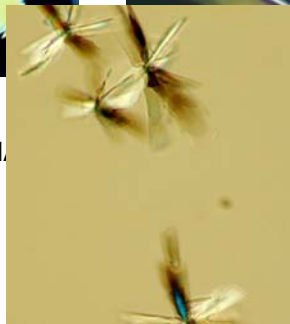
X2-F1

d l-amphetamine / H3PO4 /H



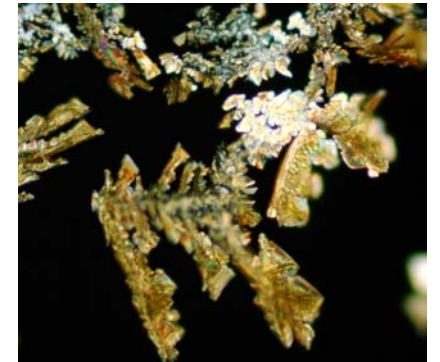
X2-F2

ine / H3PO4 /HAuCl4



X2-G1

2,3-MDMA / dil. HCl /HAuCl4



X2-H1

2,3-MDA / dil. HCl /HAuCl4

X2-J1

MDPA / dil. HCl / HAuCl4



X3-A1

methamphetamine / HCl / platinum chloride



X3-B1

cocaine / HCl / platinum chloride



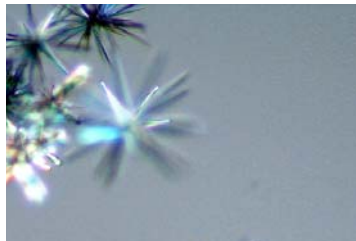
X3-B2

cocaine / HCl / platinum chloride



X3-B3

cocaine / HCl / platinum chloride



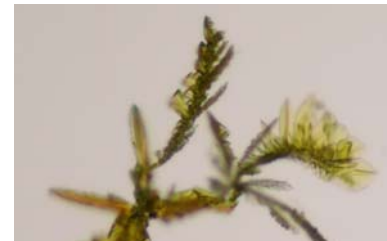
X26-A1

3,4-MDA / HCl / platinum chloride



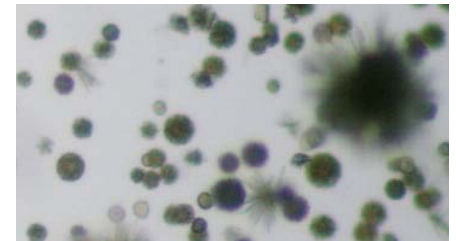
X26-A2

3,4-MDA / HCl / platinum chloride



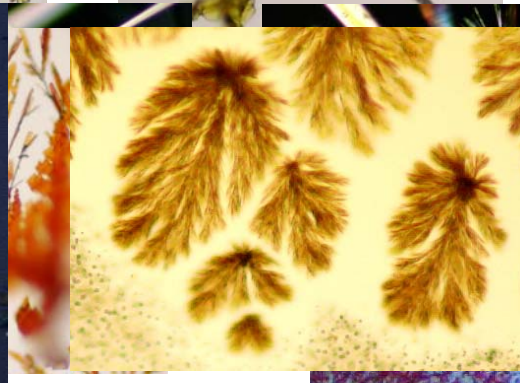
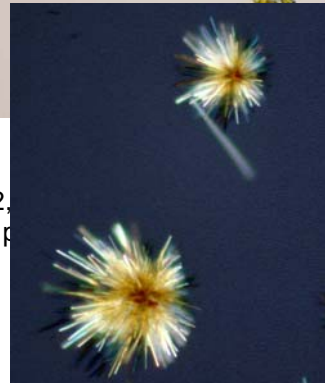
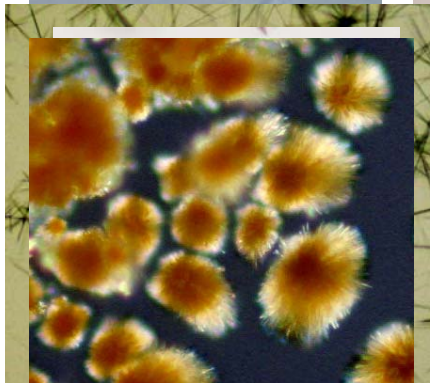
X26-B1

3,4-MDA / HCl / platinum chloride



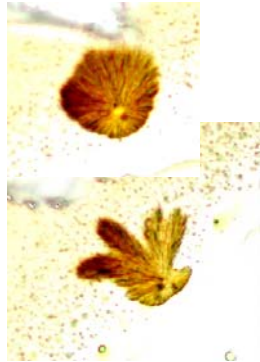
X26-B2

3,4-MDA / HCl / platinum chloride



X26-B3

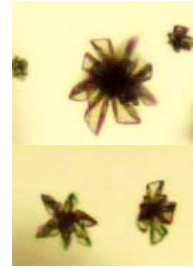
3,4-MDA / HCl / platinum chloride



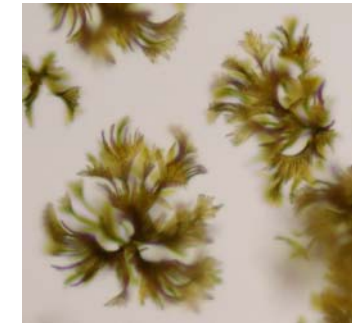
X26-B3
hydrocodone / platinum bromide



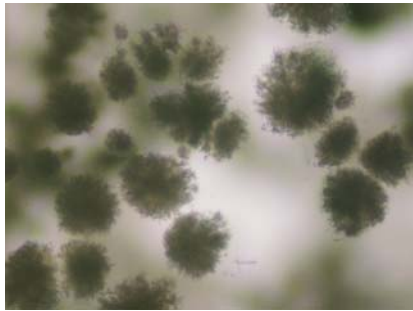
X26-C1
ketamine / platinum bromide



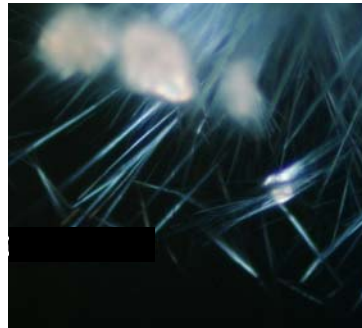
X26-D1
Methadone /
dil. HCl / platinum bromide



X26-E1
2,3-MDMA / platinum bromide



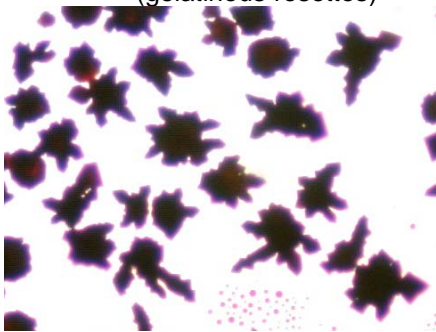
X Marme-A1
codeine / dil. HCl / Marme's
(gelatinous rosettes)



X Marme-B1
morphine / dil. HCl / Marme's



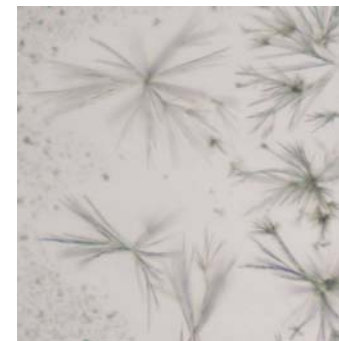
X Cu(NO₃)₂/AgNO₃-A1
GHB / Cu(NO₃)₂/AgNO₃



X Ptl-A1
ketamine / platinum iodide



X Hgl-A1
heroin / mercuric iodide



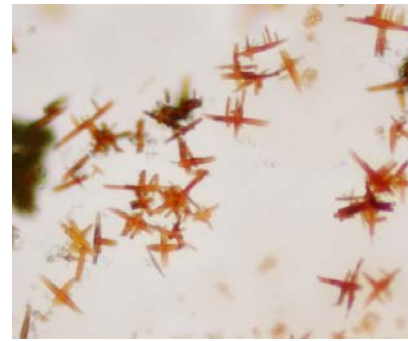
X HgCl-A1
methadone / mercuric chloride



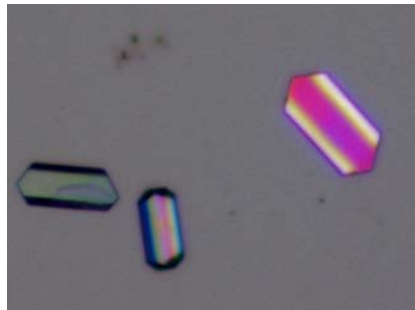
XAuBr-A1
2,3-MDA / HAuBr₄



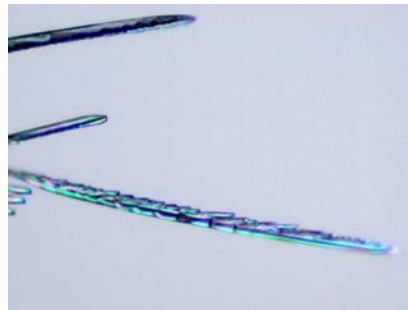
XAuBr-B1
3,4-MDA / HAuBr₄



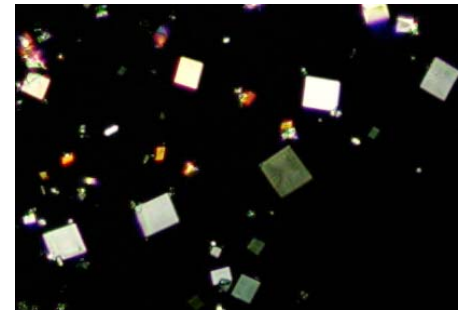
XAuBr-C1
3,4-MDPA / HAuBr₄



XNaPrus-A1
Hydromorphone /
Sodium Nitroprusside



XNaPrus-A2
Hydromorphone /
Sodium Nitroprusside



XHBr-A1
PCP / 10% HBr

6. Training Program

A. Training Outline

1. The training section is separated into modules which group together classes of narcotics and details the analyses one uses to confirm the presence of a given controlled substance within the class. The modules are:
 - a. Marijuana
 - b. Cocaine
 - c. Phenethylamines
 - d. MDA and derivatives
 - e. Hallucinogens (including LSD, psilocybin, PCP, etc.)
 - f. Opiates
 - g. Pharmaceuticals
 - h. GHB and derivatives
 - i. Analytical Techniques and Procedures

2. As a guideline, each module describing a class of narcotic may contain the following:
 - a. A fact sheet
 - b. Descriptions of presumptive tests
 - c. Descriptions of confirmatory tests
 - d. Descriptions of optional tests
 - e. Training exercises
 - f. Proficiency test requirements
 - g. Courtroom tips
 - h. A list of references
 - i. Copies of references

B. Training Manual

1. Contents of the training manual are prepared in a separate document. Please refer to it for specifics about the Narcotics training plan
2. Modules which supplement the training manual are also provided under separate cover. Not all training sections have a training module.