SAN DIEGO POLICE DEPARTMENT
FORENSIC SCIENCES SECTION

FORENSIC CHEMISTRY UNIT

NARCOTICS ANALYSIS METHODS MANUAL

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1.0 GUIDELINES/POLICIES

1.1 GENERAL GUIDELINES

1.1.1 This manual covers methods and procedures utilized in the analysis of the typical drugs of abuse that comprise the majority of this section's narcotics casework. It is not meant to cover rare and infrequent submissions. In the case of a non-routine submission, the criminalist, using sound scientific principles, will select an examination scheme comprised of the types of analyses available to them and outlined in this manual. Standards, controls, and reference materials must be fully documented in the case packet. In the event that new procedures, methodology, or instrumentation must be utilized in an analysis, the new method must be validated and approved in accordance with laboratory procedures prior to use.

1.2 ITEMS NOT EXAMINED

1.2.1 The following items are not routinely analyzed because the DA's office will not file on these cases. For unusual cases, however, requests for analysis will be considered on a case-by-case basis by the unit supervisor.

1.2.1.1 Syringes
1.2.1.2 Drug paraphernalia
1.2.1.3 Residue quantities and quantities weighing less than 0.04 grams
1.2.1.4 Marijuana and Marijuana products, including concentrates and edibles, only cases
1.2.1.5 Impounds without subject's name or identifier (except "buy" cases)
1.2.1.6 Liquid other than suspected PCP or GHB type compounds of less than approximately 0.5ml
1.2.1.7 Precursors and breakdown products of controlled substances unless they are the only chemicals present

1.2.2 Items that pose a safety hazard to lab personnel, such as suspected terrorist powders, possible explosives, and materials that react with strong acids and bases used in chemical testing, will not be analyzed.

1.3 DRUG ANALYSIS

1.3.1 Only one case shall be open in the criminalist's work area at a time.

1.3.2 All impounds will be thoroughly inventoried.

1.3.3 Sampling Plan: The number of items analyzed is dependent on the charges filed in the case, such as possession or sales related charges.

1.3.3.1 The casework approach for each criminalist with respect to items tested will be to analyze every appropriate item in the submission, or in the case of larger submissions, to analyze enough items of evidence to meet the charges. For possession cases the criminalist will test one of each drug type. For cases in which numerous items were submitted for examination, the criminalist will test and report on enough items to fulfill possession for sales criteria for that particular drug. There is not a minimum number of items for examination.

In general for sales cases, 60% of each drug type will be analyzed per suspect. Each criminalist will keep in mind that enhancements exist for possession of certain weights of cocaine and methamphetamine of 1 ounce, 2 ounces, kilos, and so on, and ½ ounce increments for heroin that may require more analysis.

1.3.3.2 The number of items actually tested will be up to the criminalist and will be reflected in the narcotics report, along with the analytical results and weight for those items. No assumptions will be made in the notes or final written product as to the contents of any untested submissions.

1.3.4 Each criminalist must determine the appropriate tests to use based on the type of suspected drug.

1.3.5 If after performing color or instrumental tests a criminalist decides not to conduct further testing on an item, provided that controlled substances are being reported for that incident and individual, the criminalist may write “initial exam only” (IEO) in their notes and stop testing. This does not need to be written in the printed report. This can also be done with federally controlled or non-controlled medication with a visual inspection or preliminary identification. If it is the only item, the report will reflect the apparent visual identification with no further examination required.

1.3.6 Chemical analysis may be done in the hood or at the criminalist’s laminar flow station. Crystal tests may be done at the criminalist’s laminar flow station.

1.3.7 The base form of cocaine is distinguished from the salt.

1.3.8 Suspected PCP and Fentanyl cases along with other possible hazardous samples must be handled with gloves and be kept in the hood during analysis. Eye protection is recommended.

1.3.9 Analyzed syringes will not be recapped by criminalists. After removing the cap and dispensing a portion of the contents for analysis, the uncapped syringe will be replaced to the safety tube using a one handed technique.

1.3.10 If multiple of the same drug type are present, a criminalist may test multiple items to meet the minimum weight requirements of 0.04 grams or greater.

1.3.11 If an criminalist is unavailable for court, the unit supervisor will assign another criminalist to reanalyze the case.

1.4 CONSUMING SAMPLES FOR ANALYSIS

1.4.1 Occasionally analysis is required on small or trace level samples where the sample may be consumed in analysis. In these instances, the unit supervisor is notified. In addition, permission to consume the sample must be obtained from the DA assigned to the case. If a DA has not been assigned to the case, the detective assigned to the case can grant permission to consume the sample. This process should be documented in the case notes.

1.4.2 After obtaining permission to consume the samples, criminalists should save any remaining extracts. These extracts would be available should additional work be required by the original criminalist, another criminalist if necessary, or a defense expert. Extracts can be placed in micro centrifuge tubes and maintained with the original impound. Notes will indicate how the sample was prepared and maintained. All extracts will be appropriately labeled. Refer to the laboratory policy statement in the quality assurance manual.

1.5 MARKING ANALYZED ITEMS

1.5.1 Individual containers housing analyzed items must be identified to include:

1.5.1.1 Barcode number
1.5.1.2 Initials

1.5.2 If multiple solid substances are housed in the same container, the criminalist must put some marking on the specific substance analyzed.

1.6 ACCEPTABLE CRITERIA FOR PRELIMINARY REPORTS

1.6.1 Preliminary testing will be performed on powders, liquids, solids, unidentified pills, etc. for use at preliminary hearings. If a case goes on to trial, a final report will be prepared.

1.6.2 In general, most cases, with the exception of pharmaceutical preparations and odd drugs, will be preliminarily analyzed by at least two independent tests. These include a combination of two of the following:

1.6.2.1 Color tests
1.6.2.2 Crystal tests
1.6.2.3 Instrumental tests
1.6.2.4 Microscopic examination (Marijuana\concentrated cannabis, and mushrooms)

1.6.3 Preliminary analysis can consist of only instrumental testing without microscopy, or color and crystal tests.

1.6.4 The criminalist may only report out results for those items that have been analyzed.

1.6.5 All reports will be technically and administratively reviewed prior to release.

1.7 ACCEPTABLE CRITERIA FOR FINAL REPORTS

1.7.1 All final examinations, unless specifically exempted in this manual, will require a minimum of an identifying crystal test and instrumental test or two instrumental tests.

1.7.1.1 If standards are not available for GC retention time comparison without another instrumental test, and a crystal test is not available for identification, the MS data alone can be used for identification. If identification of an unknown substance is made using only the MS data, the final result will be reported as "MS only" indicating that a retention time identification was not made.

1.7.2 The criminalist may only report out results for those items that have been analyzed.

1.7.3 All reports will be technically and administratively reviewed prior to release.

2.0 PROCEDURES FOR COLOR TESTS

2.1 COLOR TESTS (General Procedure)

2.1.1. Transfer small portions of the sample to the depressions of a white spot plate as needed.

2.1.2. Transfer drop-wise volumes of each appropriate color test reagent(s).

2.1.3. Mix regent(s) and sample with a plastic disposable pipette when necessary.

2.1.4. Allow appropriate time to observe any color reaction. Note reaction on analysis page.

2.1.5. Run blanks when appropriate.

2.1.6 A standard may be run for comparison.

2.2 COLOR TESTS (Drug Specific Procedures)

The results of color tests are presumptive and can direct subsequent confirmation testing. The number of color tests used is at the discretion of the criminalist and is dictated by the form of the substance. In general, a full set of color tests is not applied by the criminalist. The color tests in bold are the ones typically used for color testing on suspected drug substances.

2.2.1 Amines

Amphetamine:

Wagner-	No Reaction
Marquis-	Orange or Orange → Brown
Nitroprusside-	No Reaction
Liebermann-	Red → Orange

Methamphetamine:

Wagner-	Brown Precipitate
Marquis-	Orange or Orange → Brown
Nitroprusside-	Blue
Liebermann-	Orange

MDMA:

Wagner-	Brown precipitate
Marquis-	Green and/or Purple → Black
Nitroprusside-	Blue
Liebermann-	Orange/Brown/Black Brown
Mecke-	Green/Blue

MDA:

Wagner-	Brown Precipitate
Marquis-	Green and/or Purple → Black
Nitroprusside-	No Reaction
Liebermann-	Orange/Brown
Mecke-	Green/Blue

2.2.2 Cocaine and Cocaine Base

Wagner/H_2O-	Brown Precipitate for Cocaine
	No reaction for Cocaine Base
Wagner/H_2O/HCl	Brown Precipitate for Cocaine Base
Marquis-	No Reaction
CoSCN-	Blue Particles

2.2.3 Fentanyl

Wagner-	Brown Precipitate
CoSCN-	Blue on Particles
Marquis-	Weak Orange (sometimes)

2.2.4 LSD

Van Urk Color Test-	Purple

(Note: Add 1-2 drops of Van Urk and an equal volume of Conc. HCl)

Note: Filter Paper Analysis
Extract the LSD with methanol or chloroform or with a 3:1 combination respectively by placing the LSD onto a piece of filter paper. Then add the solvent drop by drop to the LSD causing the LSD to elute out from the center of the filter paper towards the edges. Check for extracted LSD by both long and short UV fluorescence using a pencil to outline the active areas. Apply the Van Urk reagent to different active areas in the presence of dry heat. Run an appropriate negative control on the same filter paper.

2.2.5 Opiates
Heroin

Wagner-	Brown Precipitate
Marquis-	Purple
Mecke-	Green

Codeine

Wagner-	Brown Precipitate
Marquis-	Violet
Mecke-	Blue/Green

Morphine

Wagner-	Brown Precipitate
Marquis-	Purple
Mecke-	Green/Blue

2.2.6 Phencyclidine

Wagner/H20-	Brown Precipitate
Wagner/HCl-	Brown Precipitate
CoSCN-	Blue

2.2.7 Steroids
Colors

Sulfuric Acid Based Reagents-	Unusual Vibrant

2.2.8 GHB

Ferric Chloride Solution-	Reddish Orange
Duquenois Reagent #1/Chens # 2-	Blue/Green
Chens #2-	Blue
CoSCN-	No Reaction
+HCl-	No Reaction
Lieberman's-	No Reaction

GBL

Ferric Chloride Solution-	No Reaction
Duquenois Reagent #1/Chens # 2-	No Reaction
Chens #2-	No Reaction
CoSCN-	Blue
+HCl-	Light Green
Lieberman's-	No Reaction

1, 4 Butanediol

Ferric Chloride Solution-	No Reaction
Duquenois Reagent #1/Chens # 2-	No Reaction
Chens #2-	No Reaction
CoSCN-	No Reaction
+HCl-	No Reaction
Lieberman's-	Fizzy Purple

3.0 PROCEDURES FOR CRYSTAL TESTS

3.1 CRYSTAL TESTS (General Procedure)

3.1.1 Transfer a small portion of the sample for analysis.

3.1.2 Add a small drop of the reagent(s) needed to produce crystals.

3.1.3 Mix the sample and reagent, if needed, with a toothpick.

3.1.4 Immediately view and record the results of the crystal formation.

NOTE: See the drug specific procedures for specific crystal test information.

3.2 CRYSTAL TESTS (Drug Specific Procedures)

3.2.1 Amines

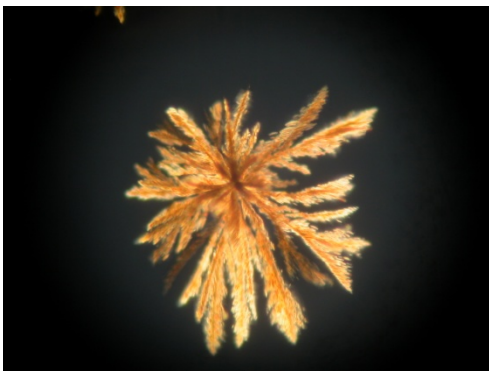
3.2.1.1 Amphetamine/Methamphetamine- Gold Chloride/Phosphoric Acid (Hanging Drop Crystal Test)

Amphetamine ***Methamphetamine*** ***DL-Methamphetamine***

Place a small portion of the sample in a spot plate well. Add a drop of saturated NaOH to the well and place a drop of gold chloride/phosphoric acid reagent to a microscope slide. Invert the microscope slide over the spot plate well to expose the fumes produced from the sample to interact with the reagent on the slide. Allow approximately 2 minutes for the fumes to react with the reagent, invert the slide, and examine microscopically. Amphetamine produces fan-shaped crystals. Methamphetamine produces clothespin-shaped crystals. DL-Methamphetamine produces crystals that appear as clothespin crystals linked back-to-back (see photos above).

3.2.1.2 MDMA Gold Chloride (Direct)

MDMA



Place a small amount of material on a slide. Add a small amount of reagent to the material on the slide and examine microscopically for gold-colored crystals that are a 3-dimensional maple-leaf shape (see photo above).

3.2.2 Cocaine and Cocaine Base - Gold Chloride (Direct)

Cocaine

Place a small amount of material on a microscope slide. Add 1 drop of 0.5N HCl to sample and mix. Place one drop of aqueous gold chloride near mixture, stir together and examine microscopically for feathered X-shaped crystals (see photo above).

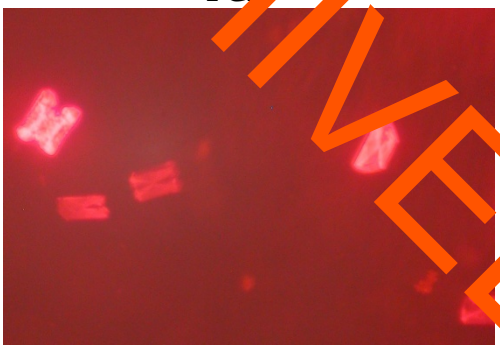
3.2.3 Heroin- Mercuric Iodide

Heroin

Place a small amount of material on a microscope slide. Add 1-2 drops of Mercuric Iodide reagent. Examine microscopically for gold-colored crystals with blue blades (see photo above). The slide can be streaked with a toothpick to help stimulate crystal growth, and crystals often take several minutes to grow.

3.2.4 PCP- Potassium Permanganate ($KMnO_4$)

PCP



Place a small amount of material on a microscope slide. Add a drop of 0.5N HCl or water to the material on the slide. Add a few small $KMnO_4$ crystals to the slide and mix to start crystal growth. Immediately examine microscopically for pink bow-tie shaped crystals (see photo above).

3.2.5 GHB- Silver Nitrate/Cupric Nitrate

GHB

Place a small amount of material on a microscope slide. Add a drop of reagent to the material on the slide and mix. Examine microscopically for the presence of gray plate-like crystals, which often are overlapping (see photo above). Crystals will often grow on the edges of the drop after about 5 minutes.

Or

Place a drop of the sample on a slide with a drop of the reagent. Combine via a "neck" by drawing one drop into the other. Crystals will grow on the edge of the drop in about 5 minutes.

4.0 IDENTIFICATION OF PHARMACEUTICAL PREPARATIONS

4.1 General Apparent Visual and Preliminary Identification Procedures

4.1.1 The following resources can be utilized for apparent identification of commonly prepared pharmaceutical preparations when the manufacturer's code/logo is clearly visible. For a preliminary identification, the type of pharmaceutical (pill/tablet), the color, shape, and logo/code must all be consistent with the reference description. For sealed pharmaceutical preparations and liquids, the label information may be used for identification.

- Drug ID Bible
- Rx-ID CD
- Ident-A-Drug
- Imprint code on the prescription label matching that visible on the tablet or capsule.
- Label information from blister packs and other sealed containers including liquids.
- On-line drug identification websites.

A photocopy or printout from a reference source is included in the case notes, when utilized for apparent tablet identification.

4.1.2 For the identification of pills and liquids in unsealed containers, an instrumental or crystal test is required.

4.1.3 If a significant physical characteristic of the questioned pharmaceutical differs from the description in the reference material, an instrumental test is required for an identification to be reported.

4.1.4 If a pharmaceutical item does not contain a controlled substance in the California Health and Safety Code, the report will reflect that a non-controlled or federally controlled medication is present. No further analysis will be performed on the item.

4.2 Final Identification Procedures

4.2.1 A final analysis requires a standard or secondary instrumental or crystal test.

5.0 GC/MS ANALYSIS

5.1 Disclaimer

The following are methods used to identify various compounds. While they are general guidelines, individual compounds may be identified under multiple methods. The method choice is at the criminalist's discretion. The parameters of the methods may be adjusted to optimize the analysis for a given compound or group of compounds.

5.2 General Sample Preparation Guidelines

5.2.1 Powder/Crystal and Solid

Place a small amount, generally about 0.01 grams or less of the material, into a test tube or injection vial and dissolve in approximately 1-1.5 ml of solvent (typically methanol or hexane). More material may be used based upon concentration. Inject the sample onto the GCMS using an appropriate method.

5.2.2 Liquid

Dilute the liquid into approximately 1 ml of methanol. The amount of liquid used is based upon concentration. Inject the sample onto the GCMS using an appropriate method.

5.2.3 Other

Dilute with 1 ml of methanol or extract the sample with the appropriate solutions/methods. Concentrate the extract if necessary and dissolve this extract into approximately 1 ml of methanol. Inject the sample onto the GCMS using an appropriate method.

5.2.4 An appropriate negative control will be run prior to each sample. Case sample results will not be accepted if the blank prior to the case sample contains identifiable peaks attributed to possible carryover or reagent contamination

5.3 Specific Sample Preparations

5.3.1 Barbiturates- Dissolve a small amount of sample into approximately 1 ml of methanol. Analyze using appropriate GC/MS method.

or

Dissolve a portion of the sample into water.
Acidify with 0.1N HCl.
Extract with chloroform.
Dry down and reconstitute with approximately 1 ml of methanol.
Analyze using appropriate GC/MS method.

5.3.2 Caines- Dissolve samples of cocaine-HCl in methanol.
Dissolve samples of cocaine base in hexane.
Analyze using appropriate GC/MS method.

5.3.3 LSD- Dissolve a small amount of sample into approximately 1 ml of methanol. Analyze using appropriate GC/MS method.

For sugar cubes, examine under UV light (both short and long wavelength). Find the most concentrated spot of fluorescence and hold this side closest to a spot well. Wash the cube with methanol dropwise over the spot well. Collect the concentrated methanol with a micropipette and place into a test tube. Dry down the concentrated methanol with air under the hood. Reconstitute with a small amount of methanol. Place the sample into a GC/MS micro vial insert, and analyze.

For blotter paper, extract directly with methanol. Allow the sample to sit in the dark for at least 20 minutes for GC/MS analysis.

5.3.4 PCP- If the PCP is contained on plant material, extract by briefly washing with methanol. Vortex the sample for 5-10 seconds and decant, after allowing the plant material to settle, into a second test tube. Analyze using appropriate GC/MS method.

If the PCP is contained in a liquid, place 1 drop of the liquid into a test tube and add approximately 3-4 ml of methanol to the tube and vortex the mixture. Transfer approximately 1 ml of the sample to a GC/MS vial and analyze using appropriate GC/MS method.

5.3.5 Steroids- Dissolve a small amount of sample into approximately 1 ml of methanol. Analyze using appropriate GC/MS method.

To clean-up oil based steroids a in a mixture of steroid/hexane/methanol (1:2:1) extraction can be used, as follows:

Vortex the oil containing the steroid and hexane mixture. If the oil and hexane mix, add the methanol to the oil/hexane mixture and vortex. The steroid will elute into the methanol. Pull off the methanol layer (top layer) for GC/MS analysis.

If the oil and hexane solutions do not mix, the steroid will elute into the hexane. Pull off the hexane layer and place in a test tube. The hexane layer may be the upper or lower layer depending on the type of oil used in the preparation. The hexane layer will be clear and colorless while the oil layer will appear somewhat viscous. If you cannot determine which layer is the hexane layer, add hexane dropwise to the mixture and observe in which layer the drop settles. Mix the hexane layer with methanol. The steroid will elute into the methanol layer. Transfer the methanol layer (lower layer) to a chromatography vial for GC/MS analysis.

5.3.6 GHB/GBL- Dissolve a very small amount of sample (less than 1 drop) into approximately 1 ml of methanol. Analyze using appropriate GC/MS method.

Note: If GC/MS confirmation is positive and if GHB crystals were produced, results are reported as GHB. If no crystals were produced during the preliminary tests, results are reported as GHB/GBL.

5.3.7 Pills- For GC/MS analysis of tablets, the following extraction process is typically used:

Pulverize the pill into a fine powder (use enough of the tablet to get approximately 1mg of the active ingredient), dissolve the material into approximately 1 ml of methanol for GC/MS analysis. Analyze the extract on the appropriate program for the type of pill suspected.

To clean out unwanted fillers, utilize the following technique: after pulverizing the pill into a fine powder, make an aqueous solution by placing the powder into a test tube and adding approximately 2 ml of water. Make the aqueous solution basic by adding 1-2 drops of saturated NaOH. Vortex the solution and add approximately 1-2 ml

of hexanes to the tube. Vortex, then allow the two phases of the solution to separate. Remove the hexanes layer and place it into a GC/MS vial for analysis. Analyze the extract on the appropriate program for the type of pill suspected, based on the initial identification using the tablet imprint/logo.

5.4 Methods

The parameters for the methods may be adjusted to optimize the analysis for a given compound or group of compounds. The current method parameters will be retained in the GCMS methods binder. The criminalist will run the appropriate method for the suspected drug.

5.5 Retention Time

5.5.1 Reference Standards

Retention times of drugs can be determined by analyzing known drug standards using the appropriate sample parameters on a GC/MS. Retention times for standards will be stable over a period of time, but must be re-established when the instrument conditions are significantly varied (change of method parameters, trimming/changing the column, etc.).

5.5.1.1 Reference spectra of drugs shall be acquired and maintained on the GC/MS. Retention time comparisons may only be used when the standard and questioned samples are run using the same GC/MS method parameters.

5.5.1.2 When there is not an appropriate laboratory standard for comparison to a questioned drug sample, other reference spectra may be utilized.

5.5.2 Identification

When the retention time of a questioned sample is being utilized as a second confirmatory test, the following applies.

5.5.2.1 Compare the retention time of the questioned sample to that of a known standard obtained in-house under the same instrumental conditions and method. The following guidelines are suggested:

Retention time of the questioned sample should be within ± 5% of the reference standard.

An ideal concentration for most compounds is indicated by a single, symmetrical, narrow peak.

5.6 Mass Spectra

5.6.1 Identification of unknown mass spectra will be made utilizing the following tools:

5.6.1.1 Reference spectra acquired on the instrument used and stored in a retrievable library (either computer or hard copy).

5.6.1.2 Reference spectra published in scientific literature/reference collections.

NOTE: An abbreviated/condensed library spectrum should only be considered a tentative identification.

5.6.2 The mass spectrum of the questioned sample must compare favorably with the standard. The principle peaks demonstrated by the standard should be present in the sample. No unexplainable significant extraneous peaks should be present. The following comments by Dr. Roger Foltz (J of Chromatographic Sciences, Vol. 12, May 1974) shall be used as a guideline.

"Agreement among the relative ion intensities within a small mass range is more important than those encompassing a wide mass range.

The higher mass ions are generally more diagnostic than those occurring at low mass (below about M/e 50).

No prominent ions in either spectrum (those with relative intensities above 10%) should be totally missing from the other spectrum, unless they can be attributed to an impurity."

5.6.3 For MSD identification the Total Ion Chromatogram (TIC), along with the mass spectra of the major peaks is evaluated.

Non-identification of peaks: the chromatogram may contain peaks inconsistent with commonly occurring controlled substances. The criminalist will examine significant peaks to determine if a commonly occurring controlled substance may be present. It is not the intent of the narcotics criminalist to identify peaks not suspected to indicate the presence of controlled substances.

5.7 Tuning Procedure for the GCMSs

5.7.1 Process

A STANDARD or AUTOTUNE is performed weekly when the instrument is in use and after any maintenance is performed on the GC/MSD.

Other options: QUICKTUNES and TARGET TUNES can be performed at the criminalist's discretion.

5.8 Requirements for Tunes

5.8.1 Tunes are to be performed weekly when instrument is in use.

5.8.2 Peak widths should be between .45 to .65 amu.

5.8.3 Peaks should be smooth and symmetrical.

5.8.4 Mass peaks should be +/- 0.2 amu

5.8.5 EM Volts approaching 3000 may indicate that preventative maintenance is needed.

5.8.6 N_2 less than 10%

5.8.21 Rel Abund: Relative abundances should be within the following limits:

69.0	70 – 100%
219.0	70 – 100%
502.0	> 1%

5.8.22 Iso Ratio: Isotope Mass Ratios should be within the following limits:

70.0	0.5 - 1.6%
220.0	3.2 - 5.4%
503.0	7.9 - 12.3%

6.0 FTIR USER GUIDELINES

6.2 Analyzing Samples

*Note: an appropriate background should be taken prior to each sample.

6.2.1 To analyze a solid sample:

Apportion just enough sample to cover at least 80% of the sample window. Avoid damaging the sample window by minimizing its contact. Run with an appropriate scan.

Or

The sample can be dissolved in an appropriate solvent prior to application onto the sample window and run as a liquid (see below).

6.2.2 To analyze a liquid sample:

Using a small disposable pipet or syringe, deliver a drop of sample in between the sample window and the crystal tip. Run with an appropriate scan.

6.2.3 To analyze a cast film from a volatile liquid sample

Using a small disposable pipet or syringe, deliver about a drop of sample on the sample window. The solvent of the sample will evaporate and leave the sample as a cast film on the sample window. Run with an appropriate scan.

6.3 Library Matches

6.3.1 Identification of unknown spectra will be made utilizing the following tools:

6.3.1.1 Reference spectra acquired on the instrument used and stored in a retrievable library (either computer or hard copy).

6.3.1.2 Reference spectra published in scientific literature/reference collections.

6.3.2 The spectrum of the questioned sample must compare favorably with the standard. The principle peaks demonstrated by the standard should be present in the sample. No unexplainable significant extraneous peaks should be present.

6.4 Printing the Spectrum

6.4.1 At this time Spectra should be printed in either black or dark blue, and at a line thickness of 3 or 4 to provide the best resolution when they are scanned for record retention. Requirements are subject to change.

6.5 Additional Information

6.5.1 The spectrum is recorded from 4000 – 650 cm^{-1}, minimum of 16 scans are used per sample, and the time the last background sample was run is automatically included in the FTIR report generated for the case packets.

7.0 Raman User Guidelines

7.1 Ready the Instrument For Use

7.1.1 Make sure the following parameters are set in the Bench tab:

Bench
Laser: Off (must turn On before use)
Laser power: 100
Aperture: 50µm slit
Min range limit: 100
Max range limit: 2000
Beam Expander: Off

7.2 Analyzing Samples

7.2.1 To analyze a solid or liquid

Place sample into an appropriate package. The normal lab packaging such as a baggie or centrifuge tube will work. Place the sample in its package over the circular opening on the universal platform sampling accessory ensuring the great amount of sample is over the window. Run an appropriate scan.

7.3 Library Matches

7.3.1 Identification of unknown spectra will be made utilizing the following tools:

7.3.1.1 Reference spectra acquired on the instrument used and stored in a retrievable library (either computer or hard copy).

7.3.1.2 Reference spectra published in scientific literature/reference collections.

7.3.2 The spectrum of the questioned sample must compare favorably with the standard. The principle peaks demonstrated by the standard should be present in the sample. No unexplainable significant extraneous peaks should be present.

7.4 Printing the Spectrum

7.4.1 At this time Spectra should be printed in either black or dark blue to provide the best resolution when they are scanned for record retention. Requirements are subject to change.

7.5 Subtracting

7.5.1 When a sample to be analyzed is diluted by a known chemical, the chemical can be subtracted from the sample to identify the controlled substance present.

7.5.2 Open the chemical spectrum if saved or add the spectrum to the same window as the diluted sample.

7.5.3 Select both spectra in the display window and go to the Process menu and click on Subtract.

7.5.4 The Subtract window will be displayed.

7.5.5 The top spectrum should be the diluted sample. The middle spectrum should be the chemical to subtract. If not, an arrow on the right of the window will switch them. The bottom spectrum is the subtracted diluted sample.

7.5.6 The spectrum on the bottom is changed using the Factor Scale on the left to subtract out the chemical to the desired level. Once done select Add to add the subtracted sample to a new window.

7.5.7 The sample spectrum can now be processed by following the Library Match and Printing steps.

8.0 Botanicals

8.1 Marijuana/Concentrated Cannabis/THC

8.1.1 Analysis

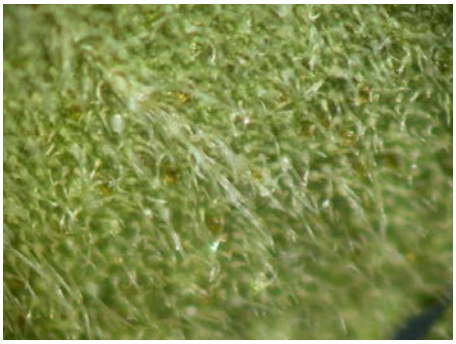
Microscopic identification of cystolithic and covering hairs is necessary for the identification of marijuana plant material. This identification requires the addition of a positive Duquenois-Levine result and identification of a cannabinoid using GCMS analysis. The identification of THC requires a positive Duquenois-Levine result and identification using GCMS analysis, or identification using GCMS analysis and a standard.

8.1.2 Microscopic examinations:

The following are the botanical features characteristic of marijuana plants/debris. One or more of these along with instrumental analysis in required for identification.

Cystolithic Hairs: Bear claw shaped with a calcium carbonate deposit at the base; unicellular, non-glandular. Located on the top side of the leaf, the stems, and sometimes on the spine. Causes a warty appearance on the top side of the leaf.

Covering Hairs: Long, thin hairs usually more abundant than the cystolithic hairs. These hairs are fuzzy in appearance and located on the underside of the leaf.



Glandular Hairs: Mushroom shaped multicellular hair that produces resin that is usually transparent and amber colored.

Seeds: Ovoid in shape with a ridge around the greatest circumference and exhibit a reticulated surface.

Concentrated cannabis (hashish): Hashish and Hash oil is a concentration of the resinous material (including cannabinoids) of marijuana. The form appears mostly as a resinous or honey-like material

8.1.3 Duquenois-Levine Color Test

Extract the cannabinoids from the material with petroleum ether and decant into a spot plate or test tube.

Evaporate the sample to dryness. Add equal volumes of Duquenois reagent and concentrated HCl respectively. A violet color should form. Add a few drops of chloroform (Levine modification) and the purple color should extract into the chloroform forming a purple or pink center for a positive identification of cannabinoids.

Note: this test can be run without the petroleum ether extraction step. However, if the result is negative, a second test with the extraction is recommended.

8.1.4 GC/MS

A small portion of the sample can be extracted with methanol to run with an appropriate GC/MS method.

Or

A small portion of the sample can be extracted with PET ether, dried down, and reconstituted with methanol to run with an appropriate GC/MS method.

8.1.5 Extraction of Cannabinoids from Food Products

Reagents:
0.2N Methanolic KOH
1.0 N HCl
10% Ethyl Acetate: Hexane

Extraction Procedure:

1. Add sample to a 15ml screw-cap conical vial.
2. Add ~4ml hexane to each sample vial and vortex until the sample is dissolved. Add ~4ml hexane to an empty vial to act as a reagent blank for this extraction.
3. Add ~4ml 0.2N methanolic KOH to each vial. Vortex briefly and shake for 5 minutes.
4. Centrifuge to separate layers. Remove and discard the top hexane layer.
5. Add ~6ml hexane. Vortex briefly and shake for 5 minutes. Centrifuge and discard the top hexane layer.
6. Repeat step 5.
7. Add ~1ml deionized water. Notice a small amount of hexane comes out of solution. Add ~6ml hexane. Vortex briefly and shake for 5 minutes. Centrifuge and discard top hexane layer.
8. Add ~1.5ml 1.0N HCl and check pH to ensure the solution is acidic.

9. Add ~3ml 10% ethyl acetate: hexane. Vortex briefly and shake for 5 minutes.
10. Centrifuge to separate layers. Transfer top organic layer to a new tube.
11. Dry down the organic layer and reconstitute with methanol.
12. Transfer to properly labeled injection vials and load samples on the GC/MS for analysis.

Reference: AFIP, Department of Defense Drug Detection Quality Assurance Laboratory: THC Quantitation in Hemp Oil.

8.2 Peyote

8.2.1 Extraction Procedure to Prepare Sample for Analysis

- Grind a button with a mortar and pestle. Freezing the sample may be necessary when attempting to grind fresh plant material.
- Make a bicarbonate slurry by adding sodium bicarbonate to the plant material and grind with the button into a paste.
- Extract into chloroform, draw off chloroform layer, and evaporate with air or nitrogen to dryness.
- Reconstitute with methanol for GC/MS analysis.

8.2.2 Color Tests

Mescaline:	Marquis-	Orange
	Mecke-	Orange → Brown
	Lieberman-	Black

Anhalomine:	Marquis-	Lavender
	Mecke-	Green → Blue

Lophoporine:	Marquis- Mecke-	Lavender Green → Blue

8.2.3 GC/MS

A good extraction to ensure a clean sample must be performed before it can be injected. Dissolve the cleaned sample in methanol. Analyze using the appropriate method. Mescaline should be identified via GC/MS. The results will be reported as "contains mescaline".

8.3 Psilocin/Psilocybin

8.3.1 A preliminary result for Psilocin/psilocybin consists of positive results for color tests and microscopic examination of spores, or a positive instrumental test. A final result for psilocin/psilocybin consists of positive preliminary result and an additional positive instrumental result.

8.3.3 Macroscopic characteristics of Psilocin/Psilocybin Mushrooms:

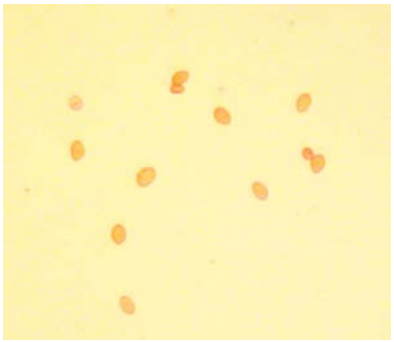
- Fluted stem.
- Inky blue coloring on various areas of the stem and where the stem joins the cap.
- Gold colored crinkled cap with dark gills underneath.

NOTE: There are many species and not all will exhibit all of these characteristics.

8.3.4 Spore Examination

Scrape the underside of a cap lightly over a microscope slide. Add a drop of H_2O and a cover slip.

Reddish-pink ovals or "footballs" should be visible microscopically.



8.3.5 Color Tests

Cut a portion of the cap and stem into small pieces with a scalpel. Apply reagent directly to the pieces of cap or stems.

Psilocybin-

Marquis	Yellow → Green/Yellow
Mecke	Green/Yellow → Brown/Green
Va Urk	Pinkish/Purple Spot
Webe	**Red**

Psilocin-

Marquis	Gree → Black
Mecke	Green → Green/Black
Weber + HCl	**Changes From Red to Blue**

Note: When conducting the weber color te , a mush oom standard must be run alongside the unknown sample.

Procedure: Add 2-3 drops to the mushroom pieces in a spot plate.
(Red = psilocybin)
Add 1-2 drops of concentrated HCl
(Blue = psilocin)

8.3.4 GC/MS

Extraction:

- Grind the mushroom material using a mortar and pestle. For fresh plant material, it may be necessary to place a mushroom cap in a plastic cup containing liquid nitrogen and allow the mushroom to equilibrate prior to grinding. Add a minimal amount of sodium bicarbonate to form a paste.

- Extract with ethyl ether into a test tube twice (approximately 5 ml)

- Evaporate the ethyl ether to dryness and reconstitute with methanol. Analyze using the appropriate method.

Note : When identification is made using GC/MS, the results are reported as “Psilocin/Psilocybin.”

8.4 KHAT

8.4.1 A preliminary result for Khat consists of a positive result for the presence of cathinonewith GC/MS analysis. A final result for Khat consists of the preliminary result and a GC/MS retention time comparison of the results between a standard of cathinone and the unknown sample.

The presence of cathinone, with or without cathine/phenylpropanolamine, indicates that the plant material is Khat and will be reported as cathinone.

If only cathine/phenylpropanolamine is found the report will reflect those substances.

8.4.2 GC/MS

8.4.2.1 Extraction

1. Use approximately 4 grams of dried material or chop up fresh material to obtain the same amount.
2. Place material in a 250 ml Erlenmeyer flask and cover with 0.2N H_2SO_4.
3. Sonicate material in water bath for 30 minutes, resulting in the generation of a brown liquid.
4. Pour off liquid through filter funnel into a second 250 ml Erlenmeyer flask and make basic with concentrated NaOH (approximately 15 drops).
5. Add approximately 30 ml of chloroform. Mix well.
6. Remove the chloroform layer (bottom) using separatory funnel or a by pipetting into large test tubes. Evaporate to dryness using air.
7. Reconstitute the dried precipitate in approximately 2 ml or less of methanol for GC/MS analysis.

Run on Amines method.

8.5 OPIUM/OPIUM POPPIES

8.5.1 Three of the five principal alkaloids found in opium (morphine, codeine, thebaine, noscapine, and papaverine) must by present before the sample can be classified as containing opium, or the poppies can be classified as opium poppies, *Papaver somniferium*.

8.5.2 Extraction Procedure for Poppies

Dried Poppies:can be ground to a powder with a mortar and pestle. Extract powder in methanol about 20 minutes for GC/MS analysis.

Fresh Poppies: can be scored to extract the sap from the pods. Dissolve the sap in methanol for GC/MS analysis. If the criminalist is unable to extract sap from the poppies, freeze dry the pods with liquid nitrogen and grind them into a powder with a mortar and pestle. Extract powder in methanol about 20 minutes for GC/MS analysis.

8.5.3 GC/MS Analysis

Run the extract on the Universal or Alkaloid Program.

9.0 MINIMUM TESTS FOR "NO CONTROLLED SUBSTANCES DETECTED" (NCSD) RESULTS

9.1 The minimum battery of tests to be performed on substances to determine that no controlled substance was detected includes, but is not limited to, the following:

9.1.1 Tests performed are based on the form of the exhibit.

9.1.1.1 **Solids:**
Wagner's
Marquis
Cobaltous Thiocyanate
Nitroprusside
Gold Chloride (Crystal Test)
Stereoscopic exam
Duquenois-Levine (if dark substance)
Ferric chloride

9.1.1.2 **Powders:**
Same as above, plus:
Mecke
Van Urk

9.1.1.3 **Liquids:**
Wagners
Marquis
Cobaltous Thiocyanate
Nitroprusside
Libermanns
Duquenois plus Chens 2
Ferric Chloride
Van Urk

9.1.1.4 **Other:**

a. If the material appears to be something such as soap or nuts, the criminalist will describe the material in their notes and report as "apparent …," and proceed with chemical testing if necessary. If chemical testing leads the criminalist to conclude that no controlled substances are present, then "No controlled substance detected" will be reported.

b. If the material is a substance which cannot be cut with a knife or does not appear suitable for chemical testing, such as a stone or piece of glass,

the criminalist will describe the item in their notes and report as “apparent …” and:

If **no testing** was attempted, “Item … is not suitable for analysis and was not laboratory examined,” will be reported.

If **microscopy** was conducted, the test performed will be included in the report. “Item … was determined by microscopy not to be suitable for analysis. No further analysis was conducted,” will be reported.

9.1.1.5 Color and/or crystal tests do not need to be performed to conclude that no controlled substances were detected if the sample is analyzed by GC/MS using the universal program. Analysis by FTIR or Raman can be used to conclude that no controlled substances were detected if analysis by FTIR or Raman identifies the presence of a non-controlled substance**.**

9.1.2 If the results of chemical testing are negative, or do not indicate the presence of a controlled substance, the report will read, “no controlled substance detected.”

9.1.4 A colored liquid may interfere with the color tests for GBL/1,4 Butanediol. Those samples will be analyzed either by a combination of GCMS and FTIR, or by GCMS alone with the inclusion of a standard.

10.0 REAGENTS

1. 0.2N H_2SO_4

Directions for Preparation

1.) Add 2.8ml concentrated H_2S0_4 to approximately 300ml of DI H_2O in a cylinder.

2.) Bring the volume up to 500ml with DI H_2O.

2. 0.2N Methanolic KOH

Directions for Preparation

1.) Dissolve 5.611g KOH into a 500ml flask containing approximately 400ml of MeOH.

2.) Bring up to volume with MeOH.

3. 1.0N HCl

Directions for Preparation

1.) Add 42ml concentrated HCl to 500ml flask containing approximately 300ml of DI H_2O.

2.) Bring up to volume with DI H_2O.

4. 0.5N HCl

Directions for Preparation

3.) Add 21ml concentrated HCl to 500ml flask containing approximately 300ml of DI H_2O.

4.) Bring up to volume with DI H_2O.

5. 10% Ethyl Acetate: Hexane

Directions for Preparation

1.) Combine 10ml ethyl acetate with 90ml hexane.

6. Chens 2

Directions for Preparation

1% CuSO4 - Dissolve 1 gram of cupric sulfate in 100ml of H_2O.

7. Cobaltous Thiocyanate

Directions for Preparation

1.) CoSCN- Dissolve 1.0 grams of cobalt acetate and 1.5 grams potassium thiocyanate in 90ml H_2O.

2.) Add10 ml of glacial acetic acid.

3.) Add 100ml of glycerin.

4.) Mix thoroughly

8. Duquenois-Levine

Directions for Preparation

1.) Dissolve 2.8 grams Vanillin and 5.8ml Acetaldehyde in 200ml of 200 proof Ethanol. Bring total volume to 400ml with additional ethanol.

9. Ferric Chloride

Directions for Preparation

1.) Dissolve 10.0 grams of ferric chloride in 100ml of H_2O.

10. Gold Chloride

Directions for Preparation

1.) Gold Chloride – Dissolve 1.0 grams of gold chloride in 20ml of H_2O
2.) 0.5 N HCl – Add 21ml of concentrated HCl to 100ml of H_2O. Bring volume to 50 ml with additional H_2O.

11. Gold Chloride/phosphoric Acid

Directions for Preparation

1.) 1+2 phosphoric acid – 6.6ml of H_2O + 13.2ml of concentrated phosphoric acid.
2.) Gold chloride/H_3PO_4 – Dissolve 1.0 grams of gold chloride in 20ml of 1+2 phosphoric acid.
3.) Concentrated NaOH.

12. Hexane: Ethyl Ether Mix

Directions for Preparation

1.) Combine a ratio of 80:20 of hexane and ethyl ether, respectively.

13. Lieberman's

Directions for Preparation

1.) Dissolve 10.0 grams of potassium nitrite in 100ml of concentrated sulfuric acid.

14. Marquis

Directions for Preparation

1.) The Marquis requires no mixing, as it is only concentrated Sulfuric Acid and 40% Formaldehyde kept in separate containers.

15. Mecke

Directions for Preparation

1.) Dissolve 0.25 grams of selenious acid in 25ml of concentrated sulfuric acid.

16. Mercuric Iodide

1) Disolve 5 grams of Mercuric Iodide in 73ml of H2O.
2) Add 27ml of concentrated HCl.

17. NaOH (Saturated)

1.) In 500ml of DI H_2O add ~5g of NaOH solid to solution and stir. Crystals should dissolve completely.
2.) Continue adding ~5 g of NaOH to the solution until crystals no longer dissolve completely.

3.) Let sit overnight, there should be a thin crystal layer settled on the bottom.

18. Nitroprusside

Directions for Preparation

1.) Dissolve 2.0 grams of sodium nitroprusside in 40ml of MeOH. Add 5ml of H_2O. Add 5ml acetaldehyde.
2.) 2% sodium carbonate - dissolve 2.0 grams of sodium carbonate in 100ml of H_2O.

19. Platinum (Platinic) Chloride

Directions for Preparation

1.) Dissolve 1.0 gram of chloroplatinic acid in 20ml of H_2O.
2.) 0.5N HCl – Add 21ml of concentrated HCl to 100ml H2O**)**. Bring volume up to 500ml with H_2O

20. Potassium Permanganate (KMnO4)

Directions for Preparation

1.) Potassium permanganate crystals
2.) 0.5 N HCl - Add 21ml of concentrated HCl to 100 ml of H_2O. Bring volume to 500ml with H_2O

21. Van Urk

Directions For Preparation

1.) Dissolve 1.0 grams of dimethylaminobenzaldehyde in 90ml of 200 proof ethanol.
2.) Add 10ml of concentrated sulfuric acid

22. Wagner

Directions For Preparation

1.) Dissolve 1.27 grams of iodine and 2.75 grams potassium iodide in 5ml of DI H_2O.
2.) Bring volume to 100ml with DI H_2O.

23. Weber

Directions For Preparation

1.) Dissolve 1-2 grams of Fast Blue B in about 3ml of H_2O. Color should be light straw. This solution should be made fresh.