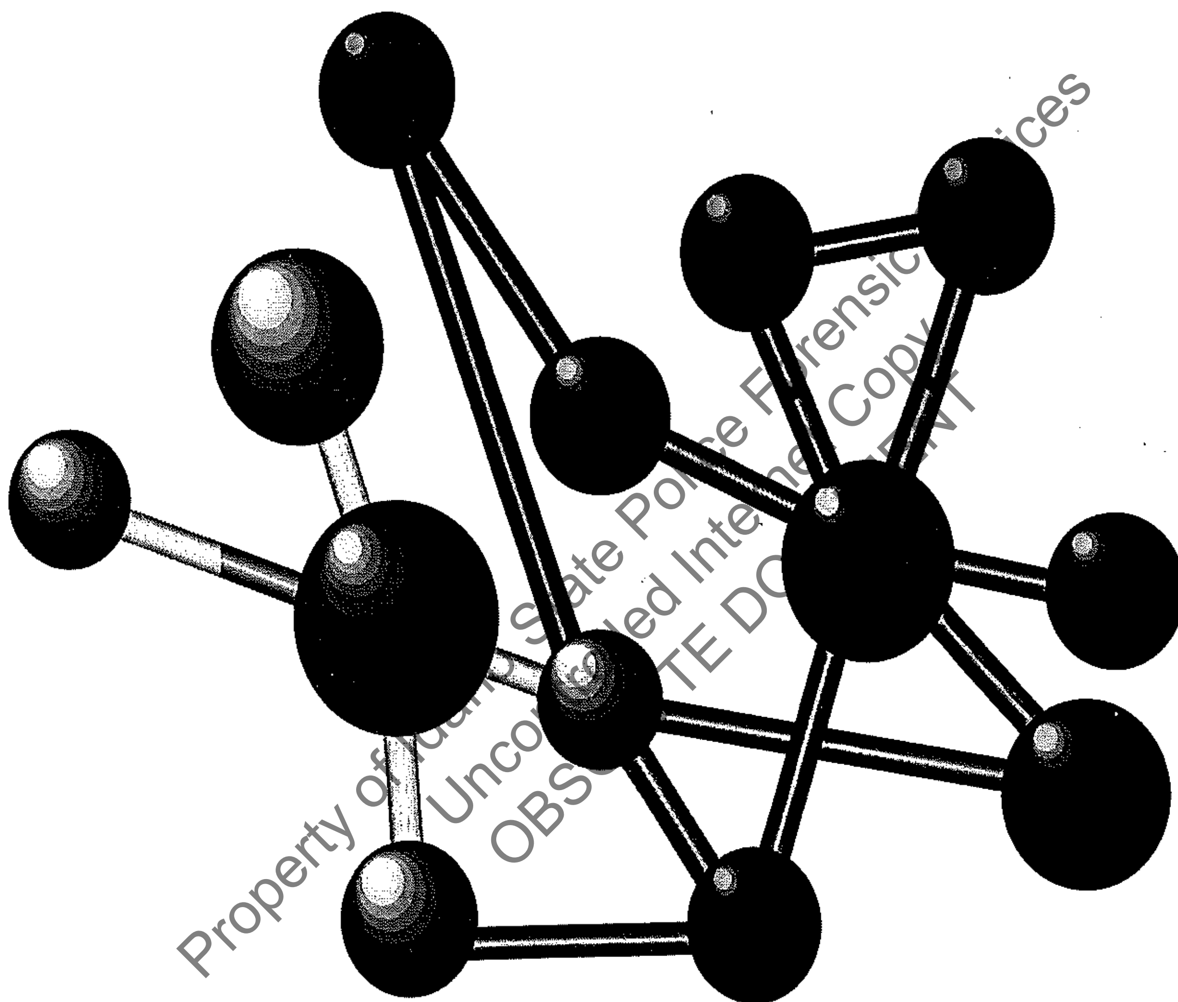
IDAHO DEPARTMENT OF LAW ENFORCEMENT BUREAU OF FORENSIC SERVICES



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DRUG PROCEDURE MANUAL

DRUG PROCEDURE MANUAL

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INTRODUCTION

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INTRODUCTION

CHEMISTRY ANALYTICAL TECHNIQUES

The following analytical techniques are currently available and utilized for sample identification and comparative analysis within the Bureau of Forensic Services.

- **Physical Characterization**

  Weight or mass determination (top-load/analytical balance)
  Visual assessment (stereomicroscope)
  Visual identification of pharmaceuticals

- **Chemical Screening Tests (Spot Tests)**

- **Microscopy**

  Compound light (polarized light microscopy)

- **Chromatography**

  Thin layer chromatography
  Gas chromatography

- **Infrared Spectroscopy**

  FT-IR
  FT-IR with microscope

- **Mass Spectrometry**

  Gas chromatography / mass spectrometry

The preceding list only shows techniques currently available "in house" in the Bureau of Forensic Services; however, we are not limited solely to these techniques. Additional instrumentation has been used and is available at other laboratories.

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PHYSICAL CHARACTERIZATION

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PHYSICAL CHARACTERIZATION

A thorough physical characterization of a material submitted for analysis includes the following observations:

- **Visual examination and physical characterization**

    Type of material: powder, liquid, tablet, plant, etc..
    Color
    Size, shape
    Morphology
    Significant markings (logos on pharmaceuticals)

    References such as The Drug Logo Index and The Physician's Desk Reference are valuable tools to help identify the contents of legitimate pharmaceutical preparations.

    Note: The use of a stereomicroscope can assist in the characterization of the above items. Sample magnification up to 40X is particularly useful in assessing plant morphology, crystalline structure and degraded samples.

- **Mass determination**

    Generally mass is determined to the 0.01 gram using a topload balance or an analytical balance can be used to weigh to 0.01 milligram. The precision of the measurement depends on the analytical balance used.

    The balance must always be clean and zeroed prior to any sample weighing.

    Determination of the net mass is made by placing the contents of an item (without the packaging) either directly on the balance or in a clean, tared weighing container.

    Actual balance readings are recorded and not rounded.

    Masses less than 0.1 gram may be reported as such.

    Masses significantly less than 0.1 gram may be reported as "a residue" or "a trace".

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CHEMICAL AND PHYSICAL SCREENING TESTS

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CHEMICAL SCREENING TESTS

Introduction

Chemical color tests are non-specific screening tests that provide preliminary data as to the nature of the substance to be identified. Certain compounds or classes of compounds produce distinct colors when brought into contact with various chemical reagents. These simple reactions can indicate the presence of a particular functional group or molecular moiety.

There is always a certain amount of subjectivity that must be taken into account when a color is reported. It is not uncommon for two analysts to describe the same color differently. Aside from the differences in reporting colors that can be attributed to the analyst, colors can also be influenced by the concentration of the sample in the reagent, by the presence of contaminants, or by the age of the reagent. Also, the length of time during which the colors are observed may influence the color reported because color transitions and instabilities are not unusual. Allowances should, therefore, be made for these differences, especially with evidence items where neither the concentration of the chemical nor the presence or composition of any contaminant is known.

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Procedure

The spot tests are conducted by placing a drop of the chosen color reagent into the well of a clean spot plate. Next, using a clean spatula, place a small amount of the substance to be tested into the drop of color reagent. Any color which is observed is noted. The purpose of placing the color reagent into the well prior to the sample is to ensure that the spot plate is clean and free of contamination.

CHEMICAL SCREENING TESTS (CONT.)

- **Reagents**

The following is a list of color test reagents commonly used in the Bureau of Forensic Services. The reagent formulations and their use can be found in the following reference in Isolation and Identification of Drugs, edited by E.G.C. Clarke.

Chen's
Cobalt thiocyanate
Dillie-Koppani
Duquenois-Levine
Formaldehyde-Sulfuric acid
Fröhde
Liebermann
Mandelin
Marquis
Mecke
Methyl benzoate
P2P spot test
Sanchez
Sodium Nitroprusside (2° Amines)
Sodium Methoxide
Sulfuric acid
Weeber
Zwikker

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Microscopy

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POLARIZED LIGHT MICROSCOPY
(Microcrystal Examinations)

Introduction

Microcrystal tests are chemical-precipitation tests in which a polarizing light microscope is used to identify specific crystalline formations. Microcrystal tests work well for many drugs and compounds containing basic nitrogen.

Procedure

Usually, the tests are performed by placing a minute amount of the sample on a microscope slide (in some cases, the sample must be dissolved in a solution such as dilute acid) and adding a drip of the reagent to it. A second technique is the "hanging drop" method where a drop of the reagent is placed on a cover slip suspended over a solution of the unknown that has been placed in a well of a microscope slide or spot plate. The solvent used in the well of the microscope slide is chosen so that the sample is volatilized. In both methods, the resulting precipitates (crystals) are then observed using the polarizing microscope at a suitable magnification.

Advantages and Limitations

Microcrystal tests are generally very sensitive, require very small sample amounts and in some cases are highly specific for a certain drug. The tests can also be the best or only practical means for determining a specific enantiomer of an enantiomeric pair. Crystal tests are limited because assessment of crystals is subjective and hard copy results (photographs)are not practical in most instances. Some crystal tests are not very specific for certain classes of drugs (i.e., opium alkaloids). Excipient material often distorts crystal formation and structure. Many of the reagents are hazardous or created toxic waste disposal problems.

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Reagents

The following are some of the more common crystal reagents and their formulations:

- ☞ Gold Chloride/HOAc - 1 gram $HAuCl_4$ dissolved in 20 ml of 20% acetic acid.
- ☞ Gold Chloride/H_3PO_4 - 1 gram $HAuCl_4$ dissolved in 20 ml 1:2 phosphoric acid in water.
- ☞ Gold Bromide - 1 gram $HAuCl_4$ dissolved in 1.5 ml 40% HBr, 8 ml H_3PO_4, and 40 ml glacial acetic acid.
- ☞ Platinic Chloride - 1 gram H_2PtCl_6 dissolved in 20 ml water.

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CHROMATOGRAPHY

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CHROMATOGRAPHY

Introduction

Chromatography is an analytical tool used to separate mixtures into individual compounds for identification by other methods. Compounds are separated based on differences in their interaction between a stationary phase (solid or liquid coating on columns or plates) and a mobile phase (gas or liquid). Identification of compounds may be performed by comparison of retention data with standards, and with detection or visualization techniques such as IR, MS, etc.

Procedure

Chromatography requires that a sample be dissolved in an appropriate mobile phase.

In thin-layer chromatography (TLC) the sample in solvent is placed on a TLC plate (a solid support e.g. glass, aluminum, plastic etc. coated with an adsorbent) and then developed in a tank containing an appropriate mobile phase (solvent system) to effect a separation of components. Individual components are visualized by observing the plate under UV light, by spraying the developed TLC plate with a chemical or by exposing to iodine vapors that reacts with the individual components to produce a colored spot.

For gas chromatography (GC) the sample is injected through a heated zone (to volatilize the sample) onto a column (usually fused silica with a liquid stationary phase coated or bonded to the inner wall) and eluted using a gas (He, N_2, etc.) as the mobile phase. Separated components of the mixture are identified, as they elute, by flame ionization detection (FID), MS, or other detection systems. Not all compounds lend themselves to GC analysis due to poor volatility or thermal degradation. Derivatization can be used to improve the chromatography of such a compound, and can also give additional data for characterization and identification.

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CHROMATOGRAPHY (cont.)

- **Calibration and Maintenance**

Retention data for chromatography must be determined for each individual system by running standards. Mechanical maintenance of GC systems are conducted as per operation manual suggestions and mobile phase supplies are replenished as needed. TLC plates are discarded after each use and TLC solvent systems are replenished when necessary. Computer data collection systems acquire data in a form which can be used by the analyst. The data printed shall reflect the data acquired.

- **Advantages and Limitations**

TLC is a rapid and inexpensive screening method which can be used as a comparative technique. TLC requires virtually no sample preparation and uses minimal amounts of sample. The technique can also be used as a method of separating compounds for further analysis (preparatory TLC). No specific structural identification of any compound can be obtained from TLC. UV light may be used for visualization of some thin layer systems as certain chemical compounds fluoresce under UV light.

GC also requires little sample preparation and minimal sample quantities. GC in combination with MS detection provides structural information for individual components. This structural information will not be valid unless individual components have first been separated by the chromatography. GC can not be used for components which are thermally labile or can not be volatilized. GC in combination with most detection systems is a destructive method.

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INFRARED SPECTROSCOPY

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INFRARED SPECTROSCOPY

Introduction

Infrared spectroscopy (IR) is an analytical technique that provides structural information enabling both characterization and identification of substances. It is based on the absorbance of infrared light energy corresponding to the quantized vibrational and rotational transitions within the molecules of a sample. Absorbance patterns can be compared to standard spectra of known compounds to identify a specific substance or identification can be made of individual chemical functional groups from portions of the absorbance pattern.

Procedure

Solid samples may be examined directly using a diamond anvil cell (DAC), attenuated total reflectance (ATR), ground with potassium bromide (KBr) and pressed into a pellet, or using diffuse reflectance sampling (DRIFTS). Liquid phase samples may be examined using KBr, sodium chloride (NaCl), or cesium iodide (CsI) plates (for non- polar liquids) or by using ATR (for polar and other liquids). Vapor phase samples are examined using a gas cell accessory containing KBr, NaCl, or CsI windows. Microscopic samples may be examined using a microscope accessory.

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Calibration and Maintenance

Fourier transform IR (FTIR) instruments are designed with built-in diagnostics and function checks which are carried out by the instrument's computer. A pulsed laser sets the timing for the interferometer and calibrates each scan, eliminating the need for any external calibration. A scan of a standard (polystyrene) performed at least once a month will demonstrate that the instrument is operating properly. A hard copy of this data is kept in the instrument log book.

Routine maintenance as recommended by the operator's manual will be conducted on a regular basis and documented in the instrument log book. A complete maintenance procedure can be found in the instrument's maintenance and calibration record book as well as in this manual under the instrumentation section of Drug Analysis. The computer is used to acquire data in a form which can be used by the analyst. The data printed shall reflect the data acquired.

- **Advantages and Limitations**

IR analysis provides non-destructive, structural information for the qualitative identification of a wide range of substances, both organic and inorganic. Analytical strengths of IR include needing limited sample quantities for analysis and the ability to determine differences between diastereomers. Weaknesses include the need for samples to be relatively pure and the difficulty of identification of individual compounds such as enantiomer pairs and racemates or compounds in a homologous series or polymer group. The lack of spectral complexity may prevent specific identification of inorganic compounds as well.

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MASS SPECTROMETRY

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MASS SPECTROMETRY

❍ Introduction

Mass spectrometry (MS) is an instrumental technique for the identification of organic compounds. Upon introducing a sample into the MS, the sample is fragmented and ionized into charged ions by bombardment with accelerated electrons. The ions are then filtered according to their mass to charge (m/z) ratio by a scanning quadrupole magnetic field. A detector and data system (computer) record the mass and quantity of ions as the spectrometer is scanning, resulting in the generation of a mass spectrum. The sample spectrum may then be compared to reference library spectra.

❍ Procedure

The sample is introduced into the mass spectrometer through a GC equipped with a capillary column. The sample is dissolved in an appropriate solvent, injected onto the GC which separates the sample into its individual components which then enter the ion source of the MS.

❍ Calibration and Maintenance

The mass spectrometer is calibrated and tuned by either of two means, manually or by an instrumental (computer) program which adjusts the MS operation parameters to achieve certain predefined performance criteria. The instrument should be autotuned on the day of use prior to begining any analysis. Should more than one analyst be running samples during the day, the instrument need only be autotuned once during that time span. Should a analytical run cover more than a single day, the autotune acquired at the beginning of the run suffices as a check until said continuous run is completed. The tuning and calibration typically utilizeperfluorotributylamine(PFTBA), a stable compound which produces ion fragments throughout the mass range for the spectrometer. These ions are used to calibrate and normalize the instrument's operation. For some work, the instrument may be tuned and calibrated using a primary standard, in order that the instrument be optimized for a particular compound. A complete discussion on tuning is found in the manufacture's

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MASS SPECTROMETRY (cont.)

operations manuals. A complete maintenance schedule can be found in the instrument's maintenance and calibration record book as well as in this manual under the instrumentation section of Drug Analysis.

Routine maintenance is performed as specified by the operations manual at appropriate intervals and documented in the instrument log book. The computer is used to acquire data in a form which can be used by the analyst; it shall not alter the fundamental data.

Advantages and Limitations

Mass spectrometry is a highly sensitive and versatile instrumental technique for a wide variety of organic compounds. The systems can be automated to increase analytical time efficiency and assist in quantitative analysis. When coupled with GC, sample preparation can be greatly reduced and complex mixtures are readily analyzed. Limitations include complete destruction of sample upon analysis and the fact that thermally labile and non-volatile samples are not readily analyzed. Spectra from closely related compounds and isomers may be so similar that conclusive identification is not possible.

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MASS SPECTROMETRY

Introduction

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Procedure

The sample is introduced into the mass spectrometer through a GC equipped with a capillary column. The sample is dissolved in an appropriate solvent, injected onto the GC which separates the sample into its individual components which then enter the ion source of the MS.

Calibration and Maintenance

The mass spectrometer is calibrated and tuned by either of two means, manually or by an instrumental (computer) program which adjusts the MS operation parameters to achieve certain predefined performance criteria. The instrument should be autotuned on the day of use prior to begining any analysis. Should more than one analyst be running samples during the day, the instrument need only be autotuned once during that time span. Should a analytical run cover more than a single day, the autotune acquired at the beginning of the run suffices as a check until said continuous run is completed. The tuning and calibration typically utilize perfluorotributylamine(PFTBA), a stable compound which produces ion fragments throughout the mass range for the spectrometer. These ions are used to calibrate and normalize the instrument's operation. For some work, the instrument may be tuned and calibrated using a primary standard, in order that the instrument be optimized for a particular compound. A complete discussion on tuning is found in the manufacture's

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MASS SPECTROMETRY (cont.)

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DRUG ANALYSIS

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DRUG ANALYSIS
(CONTROLLED SUBSTANCES)

This section of the Chemistry Technical Procedure Manual is devoted to the analysis of drugs, including controlled substances, poisons, pharmaceuticals, clandestine lab samples and unknowns.

○ INTRODUCTION

The majority of case submissions to the Chemistry Section of the BFS involves the analysis of samples to determine the presence of a controlled substance. In these cases the primary objective of the forensic scientist is to conclusively identify any and all controlled substances in a sample or conduct sufficient analysis to determine that no controlled substances are present.

Controlled substances are those substances which are so designated either by legislation or by administrative rules of the Idaho State Board of Pharmacy. The Uniform Controlled Substances Act is contained in the Revised Code of Idaho, Chapter 27. It contains definitions as well as lists of compounds.

The Uniform Controlled Substances Act organizes the drugs into Schedules I-V which define and enumerate the substances that are controlled. In general, the schedule and quantity of the drug present determine the penalty which is assessed any person convicted under the act.

The forensic scientist has an obligation to do sufficient testing to aid the judicial system in determining to which schedule a substance belongs. This requires identification of any controlled substance present as well as determining the mass in many cases. Infrequently, the scientist may need to perform semi-quantitative analysis or identify both the controlled substance and other substances with which it is mixed.

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DRUG ANALYSIS (CONT.)

The forensic scientist must provide sufficient testing to eliminate the possibility that the positive identification of a controlled substance could be due to inadvertent contamination in the laboratory. Every safeguard must be taken to ensure that a sample is not contaminated in the laboratory with any extraneous material. The integrity of the sample shall not be compromised.

❍ QUALITY CONTROL

Quality control, quality assurance and proper evidence handling are thoroughly discussed in BFS Quality Control Manual.

❍ Note Taking

Proper note taking must accompany every case analysis from the time the evidence is removed from the vault, throughout the analysis, to the return of the evidence to the vault. Notes need to be legible and complete for they often are referred to months and even years after the analysis was performed. Proper notes should include:

- ☞ Case numbers, date of analysis and analysts initials.
- ☞ Description of evidence, seal and packaging.
- ☞ Any packaging problems or evidence oddities.
- ☞ Description of each analysis and the result.
- ☞ Sample preparation and extractions.
- ☞ Conclusions upon completion of analysis.
- ☞ All communications regarding the case.

Notes and all hard copy data (spectra, chromatograms, photos and sketches) must remain in the case file.

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○ Contamination Prevention

Contamination prevention should include the following:

- ☞ Only one item of evidence is analyzed at a time.
- ☞ Batch processes (auto sampling) require each item be secure and labeled.
- ☞ Sufficient work space must be maintained and kept clean.
- ☞ Reusable items (glassware, spatulas, scalpels, etc..) must be thoroughly cleaned prior to each use.
- ☞ Disposable items are used only once.
- ☞ Sample blanks will be run whenever appropriate. A sample blank is defined as the sample resulting from a process which exactly mimics that used in the preparation of an analytical unknown, with the exception that the unknown was not added to the process.

○ Reagents and standards

Solvents and reagents are verified by running blanks and standards to safeguard against contamination and ensure proper response.

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

- ☞ Reagent name.
- ☞ Date the reagent was made.
- ☞ Initials of person making reagent.
- ☞ Reference to recipe used.
- ☞ Initials of person verifying reagent.
- ☞ Expiration date of reagent.

The headquarters laboratory maintains primary reference standards for the majority of samples encountered in case submissions.

Secondary standards will be used as standards in case work, for verifications and calibrations.

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Instrumentation

Instrumentation will be maintained and repaired by authorized and qualified personnel.

Instruments are routinely tuned and calibrated to ensure operation is acceptable and up to specifications.

An instrument log book is maintained to document all repairs and record tunes, calibrations and backgrounds.

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VALIDATION OF QUALITATIVE DRUG ANALYSIS METHODS

This is the procedure to follow when producing a new method or a **significant variation** of an existing method.

1. Keep a comprehensive log of all steps.

2. Research the literature for existing methods.

3. Research the compound which is to be targeted in Clarke, Mercke, CRC, etc.

4. Identify all possibilities of substrate for the target compound (solid, liquid, powder, pill, paper, plant material, sticky tape, reaction mixture, discard waste, etc.).

5. Choose an appropriate confirmation method of FTIR or GCMS with appropriate parameters.

6. Devise an extraction method.

7. Test method with a known standard 5 times for a new method or a split standard 5 times in parallel with an existing method.

8. Test method on old known case material (if available) or test method in parallel with present method on split samples from cases a minimum of 10 times.

9. Test method on all possible substrates.

10. Prepare a written report of method.

11. Send the report to the other Bureau of Forensic Services Laboratories for peer review and testing, and a request for a written critique.

12. After receiving a favorable critique of the new method, request administrative approval by the Quality Assurance Manager for implementation of the method.

13. Add a copy of the new method to the Drug Procedure Manual.

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Instrumentation Quality Control Procedures

○ GC and GC/MS

GC performance (chromatography, and retention time stability) is checked by running drug standards with a hard copy of the results included in each case file. Relative retention times of samples and standards must match and mass spectrum of samples and standards must match.

A solvent blank is periodically run between case samples to insure contamination is not occurring. The number and frequency of running blanks is left to the discretion of the analyst.

The syringe is thoroughly rinsed after each injection.

All sample vials are clearly marked with item number and case number.

Unattended utilization of the autosampler on the GC/MSD require the following procedures:

- ☞ A sequence table is generated listing all samples, blanks and their position in the carrousel.
- ☞ Data file headers contain the carrousel's vial position number.
- ☞ The sequence table is checked against the vials in the carrousel prior to starting the autosample run.

Hard copy sample data are maintained in case file.

Revision ⟶ GC/MS is tuned prior to each days run (or runs) and dated tune printouts are maintained in log book.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

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Instrumentation Quality Control Procedures

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- ☞ Data file headers contain the carrousel's vial position number.
- ☞ The sequence table is checked against the vials in the carrousel prior to starting the autosample run.

Hard copy sample data are maintained in case file.

The GC/MS is tuned prior to a run and dated tune printouts are maintained in log book.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

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Infrared Spectrometer

A reference standard should be ran at least once a month to verify instrumentation performance, dated hard copies are maintained in log book.

Sample support media (KBr) are periodically checked to ensure they are not contaminated. The results are maintained in the log book.

Spectrum is sufficiently resolved for library comparison.

Hard copy sample data are maintained in case file.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

Layer Chromatography

P vision →

It is suggested that a solvent blank be included with samples on each TLC plate; however, this is left to the discretion of the analyst.

Each plate must contain a standard along with any samples.

The developed TLC plates are photocopied and maintained in case file.

Reporting

The report that is issued represents a summary of the analytical findings and identifies the controlled substance or substances found and lists the item's weight. Controlled substances are reported as named in the Uniform Controlled Substances Act.

"No controlled substances". When a controlled substance is not identified the wording of the report conclusion should accurately reflect the confidence of the analysis.

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○ **Infrared Spectrometer**

A reference standard (polystyrene)should be run at least once a week to verify instrumentation performance, dated hard copies are maintained in log book.

Sample support media (KBr) are periodically checked to ensure they are not contaminated. The results are maintained in the log book.

Spectrum is sufficiently resolved for library comparison.

Hard copy sample data are maintained in case file.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

○ **Thin Layer Chromatography**

Use of a solvent blank is left to the discretion of the analyst.

Each plate must contain a standard along with any samples.

The developed TLC plates are photocopied and maintained in case file.

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○ **Reporting**

The report that is issued represents a summary of the analytical findings and identifies the controlled substance or substances found and lists the item's weight. Controlled substances are reported as named in the Uniform Controlled Substances Act.

"No controlled substances". When a controlled substance is not identified the wording of the report conclusion should accurately reflect the confidence of the analysis.

MAINTENANCE SCHEDULE FOR NICOLET MAGNA-IR 560

Monthly Validation

1. In the COLLECT menu, go into ADVANCED DIAGNOSTICS and print a report. (If you get an error message when trying this, reboot the computer.)

2. In the ANALYZE menu, go into SYSTEM VALIDATION:

   a. Click on VALIDATE and the program will prod you through the process. (The 1.5 mm and 3.0 mm polystyrene films are in the white folder to the right of the printer.)

   b. When all the information has been collected, an OMNIC SYSTEM VALIDATION REPORT will come up on the screen. Print this.

   c. Below the REPORT button are three report styles. Change to the style on the right and press PRINT.

   d. Before closing out the program, hit SAVE and save as the first three letters of the current month plus "check.csv"; eg. marcheck.csv.

   e. Close the program and return to OMNIC E.S.P.

   f. Initial all the pages generated, three-hole punch and put them into the black binder with the other reports.

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FTIR FX40 MAINTENANCE PROCEDURE

I. WEEKLY

A. Check pens and replace as needed.

II. MONTHLY OR AFTER A MAJOR REPAIR

A. Background:
1. Collect background.
2. Save background and reload.
3. Plot background as % transmission.
4. Plot background as raw interferogram.
5. Compare with previous runs.

B. Polystyrene Film Test:
1. Collect polystyrene spectrum.
2. Plot polystyrene as % transmission.
3. Compare with previous runs.

C. Clean filter.

III. BIANNUAL OR AS NEEDED

A. Clean sample compartment.

B. Clean optical bench area.

C. Clean plotter and pen holders.

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5890/5971

GCMS GENERAL MAINTENANCE PROCEDURE

I. Daily (or as needed):

A. ATUNE. Check against checksheet with accepable results. If any result of ATUNE is out of acceptable range, repeat the ATUNE. If result is still not accetable, shut down the GCMS for servicing.
B. Empty waste vials.
C. Fill rinse vials.
D. Check in printer.
E. Check syringe and clean or replace if sticking.
F. Run STARTUP sequence with amphetamine and methamphetamine standards. Compare to previous day's results. If retention times vary by more than 0.1 minute and cannot be explained by concentration change, make a new standard and repeat. If results are still not acceptable, shut down the GCMS for servicing.

II. Weekly (or as needed):

A. Change septum every 100 samples.
B. Replace the rinse solutions with fresh solvent.

III. Monthly (or as needed):

A. Run GROB mixture (or other column efficiency check mixture) and check against previous runs. The retention times should agree within 0.5 minute. If not, run a new check mixture. If the results still do not agree, shut down the GCMS for servicing.
B. Check liner. If dirty, replace liner and o-ring as needed.

IV. Quarterly (or as needed):

A. Change pre-column.
B. Clean MSD.
C. Change gold seal.

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D. Check pump oil level. If below the mark, add more oil.
E. Replace solvent trap.

V. Semi-annual (or as needed):

A. Change column (when retention times for methamphetamine base and acylated no longer reflect concentration variation and are erratic or the concentrated and dilute acylated give reversed retention times).
B. Vacuum interior.

VI. Annual:

A. Full system check (all of above checked).
B. Full system maintenance and cleaning (all of above cleaned and replaced).

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6890/5973

GCMS GENERAL MAINTENANCE PROCEDURE

I. Daily (or as needed):

A. ATUNE. Check against checksheet with accepable results. If any result of ATUNE is out of acceptable range, repeat the ATUNE. If result is still not accetable, shut down the GCMS for servicing.
B. Empty waste vials.
C. Fill rinse vials.
D. Check in printer.
E. Check syringe and clean or replace if sticking.
F. Run STARTUP sequence with cocaine and heroin standards. Compare to previous day's results. If retention times vary by more than 0.1 minute and cannot be explained by concentration change, make a new standard and repeat. If results are still not acceptable, shut down the GCMS for servicing.

II. Weekly (or as needed):

A. Replace the rinse solutions with fresh solvent.

III. Monthly (or as needed):

A. Run GROB mixture (or other column efficiency check mixture) and check against previous runs. The retention times should agree within 0.5 minute. If not, run a new check mixture. If the results still do not agree, shut down the GCMS for servicing.
B. Check liner. If dirty, replace liner and o-ring as needed.

IV. Quarterly (or as needed):

A. Change pre-column.
B. Clean MSD.
C. Change gold seal.
D. Check pump oil level. If below the mark, add more oil.
E. Replace solvent trap.

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V. Semi-annual (or as needed):

A. Change column (when retention times for cocaine and heroin no longer reflect concentration variation or give erratic results).
B. Vacuum interior.

VI. Annual:

A. Full system check (all of above checked).
B. Full system maintenance and cleaning (all of above cleaned and replaced).

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GCMS GENERAL MAINTENANCE PROCEDURE

I. Daily (or as needed):

A. ATUNE.
B. Empty waste vials.
C. Fill rinse vials.
D. Check in printer.
E. Check syringe and clean or replace if sticking.
~~F. Run STARTUP sequence with amphetamine and methamphetamine standards.~~

II. Weekly (or as needed):

A. Change septum every 100 samples.

III. Monthly (or as needed):

A. Run GROB mixture and check against previous runs.
B. Check liner and replace liner and o-ring as needed.

IV. Quarterly (or as needed):

A. Change pre-column.
B. Clean MSD.
C. Change gold seal.
D. Check pump oil level.
E. Replace solvent trap.

V. Semi-annual (or as needed):

A. Change column.
B. Vacuum interior.

VI. Annual:

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A. Full system check.
B. Full system maintenance and cleaning.

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Methods of Analysis

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METHAMPHETAMINE AND AMPHETAMINE BY GCMS

Column: 12 meter HP ULTRA 2 or a suitable longer column.

Temperature Program: 120 degrees C, hold 3 min., ramp 10 C/min, final temp 180, hold 5 min. Methamphetamine and amphetamine may also be done by the UNKNOWN.M temperature program - 80 degrees C, hold 2 min., ramp 20 C/min, final temp 280, hold 10-20 min.

Sample Preparation: Dissolve sample in water or use aqueous sample. Make very basic with Na_2CO_3. Extract with petroleum ether or hexane. Split the extract in half and acetylate one portion with acetic anhydride.

Quality Control: Compare to standards prepared in the same way. Run these on the GCMS on the same day as the unknown sample (or within 24 hours). The retention times of the unknown must have an agreement of 0.04 minutes or less with the retention times of the standard and the ion scans must be similar.

Theory: The acetic anhydride attaches to the nitrogen of the amine group forming a derivative. The derivative has a longer retention time and extra ions of mass 100 for methamphetamine and 86/118 for amphetamine.

$$2\ C_6H_5\text{-}CH_2\text{-}CHCH_3\text{-}NHCH_3 + (CH_3CO)_2O > 2\ C_6H_5\text{-}CH_2\text{-}CHCH_3\text{-}N(CH_3CO)CH_3 + H_2O$$

$$C_6H_5\text{-}CH_2\text{-}CHCH_3\text{-}NH_2 + (CH_3CO)_2O > C_6H_5\text{-}CH_2\text{-}CHCH_3\text{-}N(CH_3CO)_2 + H_2O$$

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EPHEDRINE AND PSEUDOEPHEDRINE BY GCMS

Column: 12 meter HP ULTRA 2 or a suitable longer column.

Temperature Program: 120 degrees C, hold 3 min., ramp 10 C/min, final temp 180, hold 5 min. Ephedrine and pseudoephedrine may also be done by the UNKNOWN.M temperature program - 80 degrees C, hold 2 min., ramp 20 C/min, final temp 280, hold 10-20 min.

Sample Preparation: Dissolve sample in water or use aqueous sample. Make very basic with Na2CO3. Extract with petroleum ether or diethyl ether. Split the extract in half and acetylate one portion with acetic anhydride.

Quality Control: Compare to standards prepared in the same way. Run these on the GCMS on the same day as the unknown sample (or within 24 hours). The base retention times of the unknown must have an agreement of 0.04 minutes or less with the retention times of the standard and the ion scans must be similar. The acetylated extract must have two peaks with times longer than the base extract. Because ephedrine and pseudoephedrine have the same retention times and ion scans, the report should say "Contains ephedrine or pseudoephedrine" or similar wording.

Theory: The acetic anhydride attaches to the nitrogen of the amine group forming a derivative. The derivative has longer retention times and extra ions of mass 100/101.

2 C6H5-CHOH-CHCH3-NHCH3 + (CH3CO)2O > 2 C6H5-CHOH-CHCH3-N(CH3CO)CH3 + H2O

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LYSERGIC ACID DIETHYLAMIDE (LSD) BY GCMS

Column: 12 meter HP ULTRA 2 or a suitable longer column.

Temperature Program: 100 degrees C, hold 2 min., ramp 25 C/min., final temp 280, hold 15 min.

Splitless Method: Purge initially off, turned on at 0.75 minutes.

Other Conditions: Use a clean septum and a dedicated injection liner (silanized is best). Injector 250 degrees.

Sample Preparation: Place paper cut into small pieces in a test tube. (Put tiny pellets or microdots in test tube and grind to powder). Add enough 100% methanol to cover sample. Cork tube and allow to soak several hours (overnight is better) in a dark storage place. Remove methanol and concentrate to smallest volume in microvolume insert.

Sample Amount/Type: Confirmation has been possible on ½ square paper or one pellet. It is not recommended for sugar cubes because methanol will extract part of the sugar. If sugar is in the extract when it is concentrated, it will cause the injection syringe to seize.

Quality Control: Compare to LSD standard run on same day (or within 24 hours). Retention time of the unknown must agree to 0.1 minutes or less with the retention time of the standard. The ion scans must be similar.

Theory: LSD is not very soluble in solvents (other than weak aqueous acids). By soaking the paper for a prolonged time in methanol, the LSD slowly shifts to the solvent.

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MUSHROOMS BY GCMS

Column: 30 meter HP-5MS or a suitable substitute.

Temperature Program: 200 degrees C, hold 2 min., ramp 10 C/min., final temp 280, hold 5 min. This is a splitless method.

Sample Preparation: Grind mushrooms finely, and place in a test tube. Add enough methanol to cover well, cap and shake well. Let stand for at least 30 minutes, shake again and centrifuge. Transfer supernatant into a clean test tube and concentrate to about ½ - 1 ml. Deep freeze for 1 hour. Remove from freezer and immediately add an equal volume of acetone. Mix and centrifuge. Decant into a clean tube and concentrate to ½ to 1 ml.

Color Test: Weber Test - place some ground or crumbled mushroom in a spot plate well. Add 0.1% Fast Blue BB solution and mix. This first step should change from yellow to orange within 1 minute if psilocyn is present. Transfer the liquid to a clean well and add 1 drop of concentrated HCL. If psilocyn is present, the color should go a bright blue (final color depends on the concentration of psilocyn in the mushroom material - may see green to a dark blue).

Note: Psilocybe mushrooms have blue staining stems (bruising).

Thin Layer System: T1 (10 ml methanol with 7 drops conc'd ammonium hydroxide) will separate psilocyn, psilocybin and LSD.

Quality Control: Compare to a psilocyn standard run on the GCMS on the same day (or within 24 hours) as the unknown. The retention times should have an agreement of 0.1 minutes or less and the ion scans must be similar.

Theory: Psilocybin is psilocin with a phosphate group added. When injected into the GCMS, the phosphate group is lost and the compound is confirmed as psilocyn.

References:

Garrott, A.S., Clemens,S.R., and Gaskill, J.H. "Isolation and Identification of Psilocyn from Psilocybe Mushrooms", paper presented at the Northwest Association of Forensic Scientists, May 4, 1984.

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OTHER DRUGS BY GCMS

HEROIN
Sample Preparation: Dissolve sample in chloroform and do direct GCMS, or add water and follow the procedure for aqueous liquid from a syringe.

For an aqueous liquid from a syringe, make the liquid basic to pH 8 - 10 with $NaHCO_3$ and extract with chloroform.

Temperature Program: 200 degrees C, hold 1 min., ramp 10 C/min., final temp 280, hold 5 min.
Quality Control: Compare to a heroin standard run the same day (or within 24 hours). The retention time of the sample must agree 0.1 or less with the retention time of the standard and the ion scans must be similar.

Note: The direct chloroform extraction method will work on a trace case.

COCAINE
Sample Preparation: Make a methanol extract of the powder.

Temperature Program: 200 degrees C, hold 1 minute, ramp 10 C/min., final temp. 260, hold 2 min.

Quality Control: Compare to a cocaine standard run the same day (or within 24 hours). The retention time of the sample must agree 0.1 minute or less with the retention time of the standard and the ion scans must be similar.

Note: This method will work on a trace case.

GENERAL UNKNOWNS AND PILLS
Sample Preparation: Extract with methanol (or best solvent with pills).

Temperature Program: 80 degrees C, hold 2 min., ramp 20 C/min., final temp. 280, hold 10-20 min.

Quality Control: After a library search of the available libraries has indicated the contents, confirmation must include comparison with a standard run the same day (or within 24 hours). The retention times must agree 0.1 or less with the retention time of the standard and the ion scans must be similar. Exceptions to retention time agreement would include some members of the phenethylamine family and other drugs with a very simple ion scan.

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DRUG QUANTITATION BY GCMS

Column: 12 meter HP ULTRA 2, 30 meter HP-5MS or a suitable substitute.

Temperature Program: Choose an appropriate temperature program for the target drug.

Sample Preparation: Mix the entire sample thoroughly, using the mill if the sample contains hard lumps. Take representative samples from throughout the mixture.

1. Make a dilution of the standard to a concentration of 5mg/ml, diluting with an appropriate solvent.
2. Make an initial dilution of the unknown to a concentration of 5mg/ml, diluting with the same solvent as the standard.
3. Run the standard and the unknown on the GCMS in the same manner and compare the instrument response to each. The areas of the standard and the unknown must be within 10% of one another. If they are not, adjust the concentration of the unknown accordingly.
4. Run the standard and unknown in triplicate with a solvent blank between each.
5. Calculate the concentration of the unknown using the QUANTITATION CALCULATION guideline page.
6. Report the concentration as "approximately__%", rounding off the percentage to the nearest whole number divisible by 5 (e.g. 25, 30, 55, 90).

Quality Control: Because the MS response is not linear, the standard and unknown must have similar areas for a one-point quantitation to be effective. Copies of GCMS printouts with the areas, blanks and the calculation sheet must be included in the file notes.

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QUANTITATION CALCULATION

$$C_{sample} = \frac{\left(\dfrac{A_{sample}}{A_{standard}} \times \dfrac{V_{standard}}{V_{sample}}\right) C_{standard}}{\dfrac{\text{Total Weight Sample}}{\text{Total Volume Sample}}} \times 100$$

C_{sample}	=	Concentration of sample in percentage.
A_{sample}	=	Peak area or height of sample.
$A_{standard}$	=	Peak area or height of standard.
$V_{standard}$	=	Volume injected of standard.
V_{sample}	=	Volume injected of sample.
$C_{standard}$	=	Concentration of standard.

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STEROID ANALYSIS BY GCMS

Column: 30 meter HP-5MS or a suitable substitute.

Temperature Program: 250 deg. C for 2 min., ramp 5 C/min. To 310 deg., hold 10 min.

Sample Preparation: Choose a suitable method from "PUMPED UP - STEROID ANALYSIS" based on the type of substrate (pill, oil, powder, etc.).

Quality Control: Compare to a standard run on the same day as the unknown (or within 24 hours). The retention times of the sample must agree 0.1 minutes or less with the retention time of the standard and the ion scans must be similar.

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COCAINE BY THIOCYANATE DERIVATIVE AND FTIR

Sample Preparation: Add about 2ml cobalt thiocyanate spot test reagent to the powder. Add up to ½ ml of concentrated HCl and mix well. Extract with chloroform and dry through Na2SO4 onto KBr. Make a KBr pellet for analysis by FTIR.

Quality Control: Compare to a cocaine standard that has been similarly treated.

Theory: Cocaine forms a blue ligand complex with the cobalt thiocyanate reagent. Under acid conditions, the complex is readily soluble in chloroform.

Reference: Naylor, Jon D., Phillips, Carl R., McCurdy, Robert J., and Koers, Stephen "A Simple Procedure for the Separation and Identification of Cocaine", Midwestern Association of Forensic Scientists Spring 1975 meeting.

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dl-METHAMPHETAMINE BY PIT DERIVATIVE AND FTIR

Sample preparation: Dissolve powder in water. Make very basic with Na2CO3 and extract with petroleum ether or hexane. Dry extract through Na2SO4. Add 2 drops of phenylisothiocyanate or PIT reagent and let stand for 10 minutes. Decant solvent and wash crystals with petroleum ether or hexane. Dry crystals and make a KBr pellet for analysis by FTIR.

Quality Control: Compare to a dl-methamphetamine standard that has been similarly treated.

Notes: This method can be used to confirm a non-racemic mixture (of d-meth and dl-meth). The PIT reagent will react with d-meth, but will not form crystals. The wash with petroleum ether or hexane will remove d-meth, leaving dl-meth PIT derivative crystals behind.

Reference: Microgram, Vol. XII, No. 2 (February 1979).

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METHAMPHETAMINE OR AMPHETAMINE BY FTIR

Sample Preparation: Dissolve powder in water. Make very basic with Na_2CO_3 and extract with petroleum ether or hexane. Wash extract with water and dry through Na_2SO_4. Bubble with HCl gas and wash resulting crystals with additional petroleum ether or hexane. Dry crystals and make a KBr pellet for FTIR analysis.

Quality Control: Compare to a standard that has been similarly prepared.

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EPHEDRINE AND PSEUDOEPHEDRINE BY FTIR

Sample Preparation: Dissolve powder in water. Make very basic with Na_2CO_3 and extract with diethyl ether (best solvent for pseudoephedrine) or petroleum ether. Dry through Na_2SO_4. Bubble with HCl gas. Dry the crystals and make a KBr pellet for FTIR analysis.

If the ephedrine or pseudoephedrine is in pill form or a powder with no other organic contaminants, add methanol to crushed pills or powder and shake well. Let stand for ½ hour or longer and centrifuge. Remove supernatant and place in a clean test tube. Let stand until liquid has evaporated and make a KBr pellet with the crystals that formed on the sides of the tube for FTIR analysis. Or dry methanol extract and make a KBr pellet with the resulting crystals.

Quality Control: Compare to a standard that has been similarly prepared.

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HEROIN BY FTIR

Sample Preparation: Dissolve sample in 0.1 N HCl. (May be filtered or centrifuged). Extract with chloroform. Back-extract with water. Make basic to pH 8 - 10 with NaHCO3 or Na2CO3. Extract with chloroform and dry through Na2SO4. Bubble with HCl gas. Recrystallize with petroleum ether. Dry the resulting crystals and make a KBr pellet for FTIR analysis.

Quality Control: Compare to a heroin standard that has been similarly treated.

Notes: Precaution must be taken with the pH. If the solution is made too basic, the diacetylmorphine will break down into morphine.

Reference: "Extraction of Heroin by the Panning Technique", Vol. 12 #1 NWAFS Newsletter.

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SPOT TESTS USED BY BUREAU OF FORENSIC SERVICES MERIDIAN

Marquis

Cobalt Thiocyanate

Secondary Amine

Methylbenzoate

Liebermann's

Dille-Koppanyi

Mecke

Froehde

Formaldehyde-Sulfuric Acid

Duquenois-Levine

Weber Test

P2P Spot Test

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THIN LAYER CHROMATOGRAPHY SYSTEMS USED BY BFS MERIDIAN

Chloroform - marijuana

Petroleum Ether/Diethyl Ether (4:1) - marijuana

Acetone - LSD

T1 - LSD, psilocyn and psilocybin, other drugs

VISUALIZING SPRAYS

Fast Blue BB - marijuana

PDMAB or p-Dimethylaminobenzaldehyde - LSD, psilocyn and psilocybin

Acidified Iodoplatinate - other drugs

Fluorescamine - other drugs

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CLANDESTINE LABORATORY ANALYSIS

I. LIQUIDS

A. Characterize liquid as aqueous or solvent by miscibility test.

1. Solvent
   a. Flammable.
   b. Identify with GC, GCMS or FTIR if possible.

2. Aqueous
   a. pH < 4 - identify acid:
      $BaCl_2$ - white precipitate = sulfuric acid or sulfate ion.
         - no ppt, go to next reagent.
      $AgNO_3$ - white ppt = HCl or chloride ion.
         - no ppt, go to next test.
      Copper penny green Rx = nitric acid over 40%.
   b. pH basic - go to extraction step.

B. Check for controlled substances/precursors.

1. Solvents - concentrate and run as general unknown.

2. Aqueous - spot test with Marquis reagent.
   a. Orange to brown - extract with petroleum ether for meth, part acetylated and run on GCMS methamphetamine method.
   b. Marquis neg, Liebermann's orange - extract with diethyl ether for precursors and run on GCMS methamphetamine method.

II. POWDERS

A. pH when mixed with water.

B. Spot tests:

1. Marquis - orange to brown for methamphetamine/amphetamine.

2. Liebermann's - orange with meth, amph, ephedrine & pseudoephedrine.

C. Identification by FTIR or GCMS using appropriate method if spot test(s) is positive.

D. Spot tests negative - try to ID with FTIR or inorganic methods if possible.

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CLAN LAB SPECTRAL DATA

by Tom Keener, Chico

Over the past few years since being in Chico, I've jumped in with both feet to do my part and "do" clan labs. Aaahhh, the Central Valley, summertime, and Saranex...how they warm my heart. Anyway, unwilling (or unable) to store vast libraries of mass spectral data in my head, I've compiled all the clan lab-associated compounds that I can find into table form with their most prominent ions and Chico's GCMS retention times, if available. As I come upon older issues of Microgram, etc., I update the table. We use it as a screening tool to point us in the right direction especially when we're staring at a TIC with 10 or 20 compounds. The table is in similar format to a lot of tables I've seen but it also includes meth precursors, intermediates, byproducts, and MDA/MDMA related compounds.

For what it's worth, here it is. If anyone wants an E-copy, send me a blank floppy disk and I'll get it back to you. My thanks and apologies, to all whose work I absconded. If you have any data that would be good for me to add, or notice any problems let me know.

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PROMINENT IONS AND SOME RETENTION TIMES OF CLANDESTINE LAB-ASSOCIATED CHEMICAL

Compound	Base Peak	Add'l Major Ions* (in descending abundance)				R T (METH.A) (minutes, approx)	Ref
phenyl-2-propanone	43	91	65	92	134	2.3	1,7
norpseudoephedrine	44	77	51	78	42		1
phenylpropanolamine	44	77	51	79	42	3.4	1,7
1-phenyl-2-propanamine acetamide	44	86	43	118	91		14
amphetamine	44	91	65	42	63	2.6	1,7
3-methoxyamphetamine	44	91	78	77	65		1
1-phenyl-2-propanamine	44	91	65	42	63		14
1-phenyl-2-nitropropene	44	91	65	42	63		20
2-methoxyamphetamine	44	91	122	65	78		1
N-methyl-1-phenyl-3-propanamine	44	91	148	65	51		
N-methyl-3-phenyl-1-propenamine	44	118	42	77	115		17
4-methoxyamphetamine	44	122	121	78	77		1,7
1-chloro-1-phenyl-2-aminopropane	44	132	91	105	117		1
3,4-MDA	44	136	135	77	51	4.8	7,9
an aziridine by-product	44	147	115	70	91		17
N-acetyl-MDA	44	162	43	135	77		14
4-methyl-2,5-MDA	44	166	151	91	77		1
methcathinone (secondary byproduct)	56	58	51	77	105		21
methamphetimine	56	91	65	51	57		4
ephedrine/HI byproduct	56	267	127	140	91	3.0	7
cis-3,4-dimethyl-5-phenyl-2-oxazolidone	57	42	58	56	117	4-6.2	7,8
trans-3,4-dimethyl-5-phenyl-2-oxazolidone	57	42	58	191	56	4-6.2	7,8
phendimetrazine	57	42	85	56	60	4.1	1,7
propylhexedrine	58	41	55	42			1
1-phenyl-2-butamine	58	41	91	120			2
1-(3,4-MDphenyl)-2-butamine	58	41	136	51	77		13
N,N-dimethylphenethylamine	58	42	77				2
an isosafrole oxygenation product	58	43	134	86	192		18
3-methoxymethamphetamine	58	56	91	78	77		1
methcathinone	58	77	51	56	42		21
ephedrine/pseudoephedrine	58	77	56	42	51	3.7	1,7
3,4-MDMA	58	77	135	51	56	4.9	7,9
phentermine	58	91	42	41	65		7
methamphetamine	58	91	42	56	65	2.7	1,7
2-methoxymethamphetamine	58	91	65	56	59		1
N-ethylphenethylamine	58	91	77	105			2
4-methoxymethamphetamine	58	121	78	56	77		1
1-chloro-1-phenyl-2-methylaminopropane	58	146	105	42	77		1
N-hydroxy-3,4-MDMA	60	136	135	44	77		12
phenmetrazine	71	42	56	43	77	4.0	1,7

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Compound	Base Peak	Add'l Major Ions* (in descending abundance)				R T (METH.A) (minutes, approx)	Ref
3,4-dim[illegible]yl-5-phenyloxazolidine	71	56	42	43	77	4-6.2	7,8
4-methoxy-N,N-dimethylamphetamine	72	42	44	56	121		1
N-methyl-1-(3,4-MDphenyl)-2-butamine	72	42	57	77	135		13
N,N-dimethylamphetamine	72	42	91	44	65	3.9	1,7
N-methylephedrine	72	44	42	77	56	3.9	7
3,4-methylenedioxyethylamphetamine	72	44	77	135	73		9
N-ethylamphetamine	72	44	91	65	42		7
4-methoxy-N-ethylamphetamine	72	44	121	77	78		1
N,N-dimethylephedrine	72	77	42	44	56		1
N-methylephedrine	72	77	44	42	56	3.9	7
N-methyl-1-(3,4-MDphenyl)-2-butanamine	72	89	73	77	135		15
mephentermine	72	91	42	148	65		9
N-formylamphetamine	72	118	44	91	65		4
1-phenyl-3-propanamine acetamide	73	43	117	91	177		14
N-hydroxy-1-(3,4-MDphenyl)-2-butamine	74	136	135	58	77		13
benzyl alcohol	79	108	77	107	51		3
Leuckart amination product of P2P	84	91	42	41	65		20
nicotine	84	133	162	161	42	3.5	7,9
an oxazolidine analog	85	58	70	57	42	4.0	7
5-phenyl-2,3,4-trimethyloxazolidine	85	70	57	42	148	4.0	7,8
[illegible]opylamphetamine	86	44	91	120	162	3.7	7
N-ethyl-1-(3,4-MDphenyl)-2-butanamine	86	58	41	135	77		13
N,N-dimethyl-1-(3,4-MDphenyl)-2-butanamine	86	71	42	51	77		13
tri-(isopropylphenyl)-amine	91	41	44	119	190		4
di-(isopropylphenyl)-amine	91	44	162	119	65		4
di-(isopropylphenyl)-methylamine	91	58	176	41	90		4
phenethylamine	91	65	51	63			1
dibenzylketone (diphenylacetone)	91	65	119	118	39		1
benzyl bromide	91	65	63	92	89		3
benzyl chloride	91	65	126	63	51		3
1-phenyl-2-bromopropane	91	119	51	65	198		17
benzyl methyl ketone	91	119	160	65			4
4-chlorotoluene	91	126	63	65	89		3
a-benzyl-N-methylphenethylamine	91	132	65	223	92		7
phenylacetic acid	91	136	65	92	63	3.7	1,7
benzylacetate	91	150	65	59	92		4
benzphetamine	91	148	65	92	42		1
1-phenyl-2,3-dibromopropane	91	183	185	199	197		17
1-phenyl-2-propanol	92	91	45	65	63		17
1-(3,4-MDphenyl)-2-nitro-1-propene	103	77	160	207	102		3

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Compound	Base Peak	Add'l Major Ions* (in descending abundance)				R T (METH.A) (minutes, approx)	
1-phenyl-1,3-dibromopropane	104	183	185	199	197		17
3,4-dimethyl-5-phenyl-1,2,3-oxazolidine	105	42	56	57	77	4-6.2	7,8
methylbenzoate	105	77	134	51	50		4
1-phenyl-3-bromopropane	105	91	51	77	198		17
1-phenyl-1-propanamine acetamide	105	148	43	77	79		14
benzaldehyde	106	77	105	51	50		4
1-phenyl-1-propanamine	106	79	44	77	51		14
1-phenyl-2-nitropropene (and other analogs)	115	91	105	116	163		4
1-phenyl-2-benzyl-1-propene	115	208	91	193	179		4
trans-1-phenyl-2-benzyl-1-propene	115	208	91	193	130		4
benzyl cyanide	117	90	116	89	51		3
4-cyanotoluene	117	116	90	89	63		3
cis-1-phenyl-2-benzyl-1-propene	117	116	91	208	129		4
N-methyl-1-phenethylamine	120	42	58	77	105		22
N-methyl-1-phenyl-1-propanamine	120	42	91	77	121		17
N-methylbenzylamine	120	44	91	42	121		19
a-benzylphenethylamine	120	91	103	65	77		1
nicotinamide	122	78	106	51	50		9
guaifenesin	124	109	198	81	77	4.9	7
benzyl methyl ketone benzylimine	132	105	91	78	65		4
phenyl-2-propanone-cis/trans-enol-acetate	134	43	91	135			4
a-benzyl-N-methylphenethylamine	134	91	42	65	119		1
3,4-MDphenylacetone	135	51	77	178	43		3
3,4-MDbenzylacetone	135	43	192	91	119		3
an isosafrole oxygenation product	135	77	51	178	43		18
3,4-MDphenyl-2-propanone oxime	135	77	193	51	136		12
3,4-MDphenol acetone	135	178	77	136	79		4
cis/trans-1,2-dimethyl-3-phenylaziridine	146	105	132	91	117	2.9, 3.3	5-7
3,4-dimethyl-2,5-diphenyloxazolidine	146	147	105	77	132	6.0	7,8
an aziridine by-product	147	44	115	70	91		17
5-phenyl-2,2,3,4-tetramethyloxazolidine	148	99	190	56	42	4-6.2	7,8
2-ethyl-5-phenyl-2,3,4-trimethyloxazolidine	148	190	56	204	91	4-6.2	7,8
5-benzyl-2,3,4-trimethyloxazolidine	148	190	91	42	56	4-6.2	7,8
piperonal	149	150	63	121	65		3
bis-(2-ethyl hexyl)-phthalate	149	57	167	45	46	7.0	11
2,5-DMphenethylamine	152	137	77	181	121		16
safrole	162	104	51	77	131		3
isosafrole	162	104	103	51	77		3
2,5-Dmbenzaldehyde	166	95	63	53	151		16
2-methoxy-4,5-MDA	166	151	44	165	77		9

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Compound	Base Peak	Add'l Major Ions* (in descending abundance)				R T (METH.A) (minutes, approx)	Ref
diphenylmethane	167	168	165	91	152		4
diphenylamine	169	168	51	167	83	4.8	7
Leuckart amination product of P2P	170	44	102	69	115		20
an isosafrole oxygenation product	178	43	42	72			18
an isosafrole oxygenation product	178	134	43	148	163		18
bibenzyl	179	180	91	178	165		4
1-phenyl-1,2-dibromopropane	199	198	117	116	115		17
1,3-dimethyl-6,7-MDisoquinoline	201	202	143	200	203		10
dihydro isoquinoline(?)	203	42	175	51	77		14
1,3-dimethyl-3,4-dihydro-6,7- MDisoquinoline	203	202	175	188	174		10
triprolidine	208	209	193	278	192	7.1	9
1-(2,5-DMphenyl)-2-nitroethane	209	133	77	162	147		16
4-bromo-2,5-DMphenethylamine	230	232	77	215	217		16
1,3-dimethyl-2-phenyl naphthalene	232	215	217	108	202	6.3-6.4	6,7
1-benzyl-3-methyl naphthalene	232	217	108	215	115	6.3-6.4	6,7
a naphthalene analog	232	217	215	202	216	6.6	7
a naphthalene analog	232	217	215	202	216	6.7	7
isosafrole reaction product	295	324	173	296	265		14

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MARIJUANA ANALYSIS

I. MICROSCOPIC EXAMINATION

A. Plant material must have these characteristics:

1. Cystolithic hairs - Small "bear claw" shaped hairs on the top side of the leaves or leaf fragments, so-called because of the presence of calcium carbonate at the base of the claws.
2. Unicellular hairs - Long, fine hairs on the underside of the leaf or leaf fragment, which give the underside a lighter appearance.

B. Seeds must have these characteristics:

1. Veined shell.
2. Ridged edges.
3. Point on one end and dint on the end of plant attachment.
4. Overall appearance of a miniature coconut.

II. CHEMICAL TESTS

A. Modified Duquenois-Levine Test:

1. To a dried portion of a petroleum ether extract of the plant material, add 2-5 drops Duquenois reagent and an equal amount of concentrated HCl.
2. Wait 2 minutes and add $CHCl_3$. Shake and look for reaction.
3. If marijuana resins are present, a slow purple will form during the first step. The purple complex will transfer to the $CHCl_3$ layer in the second step. Both steps must occur for a **POSITIVE TEST.**

B. Thin Layer Chromatography (TLC):

1. Spot a portion of the petroleum ether extract on a thin layer plate.
2. Spot a marijuana standard and a petroleum ether blank on the same plate.
3. Use one of the following mobile phases:
   a. Petroleum ether/diethyl ether 4:1
   b. Chloroform
   c. Benzene
   d. Petroleum ether/methanol 95:5 (if PCP may be present)
4. Visualize with Fast Blue BB salt solution spray. The cannabinoids will form characteristic colors with the spray.

C. If a microscopic examination can't be done (pipes, hash, residues, finely ground leaf material), a Modified Duquenois-Levine and 2 TLC systems must be done.

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III. SEED GERMINATION

A. Place a known number of seeds in a moist container (e.g. petrie dish with damp filter paper).

B. Check the seeds daily for 3 days.

C. Count the number of sprouted seeds and convert to a percentage of known total..

D. Seeds are considered fertile if more than 10% sprout.

IV. REPORT OF RESULTS

A. Positive microscopic, Duquenois and TLC - report as "marijuana (non-narcotic, schedule I)".

B. No microscopic possible, but positive Duquenois and 2 TLCs - report as "resins of marijuana (non-narcotic, schedule I)".

C. Positive microscopic and TLC, but negative or inconclusive Duquenois - report as "Inconclusive for marijuana or the resins of marijuana".

V. PROBLEM SAMPLES

A. Resin obscures the leaf structure - rinse leaves with petroleum ether, dry and repeat the microscopic. The solvent will remove enough resin to make the hairs visible.

B. Positive microscopic and Duquenois, but negative or questionable TLC's - re-extract the plant material, concentrate into a microinsert and run **SPLITLESS ON** the GCMS. Do a library search of the THC peak and print the comparison results. Report as "Contains marijuana (non-narcotic, CI)".

C. THC is not found with TLC, although the other resins may be present - re-extract the plant material, concentrate into a microinsert and run **SPLITLESS** on the GCMS. Do a library search on the THC peak and print the comparison results.

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MARIJUANA TRAINING GUIDELINES

1. 2-3 hours
   a. Demonstrate analysis explaining each step.
   b. Have trainee test a sample under supervision. Have trainee explain test steps.
   c. Trainee should keep a log of training.

2. 2-3 hours
   Repeat lesson one with a different criminalist.

3. 2-3 hours
   Have trainee do analysis, explaining each step. When trainee can adequately explain each part of the test, go on to lesson 4. Otherwise, repeat lesson 3 until satisfied with trainee progress.

4. 2-3 hours
   a. Supply trainee with essential reading material and answer questions about it.
   b. Review procedures for hash and marijuana seeds.

5. 2-3 hours
   a. Answers questions about reading material.
   b. Have trainee do a case under supervision.
   c. Review chain-of custody procedures.

6. 1-2 hours
   Review trainee's knowledge through mock court.

7. 3-4 hours (recommended if available only)
   Have trainee spend time at the Department of Agriculture Seed Lab examining the available seeds. These can be compared to marijuana seeds.

8. 2-3 hours
   Have trainee do the marijuana proficiency test.

9. After successful completion of proficiency test, trainee should do 10 marijuana cases under supervision. If trainee is from another agency, supply necessary items to get started with analysis. Write a letter (memo for IBFS staff member) stating the trainee's competency.

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GENERAL DRUG TRAINING GUIDELINES

I. COCAINE

A. Spot tests.
B. Direct IR.
C. Cobalt thiocyanate derivative & IR confirmation.
D. GCMS preparation & confirmation.
E. Microcrystalline test.
F. Schedule.
G. References.

II. PILLS

A. Reference search:
   1. Contents and amount.
   2. Company.
   3. Schedule.
B. Screen, confirm or no testing.
C. How to extract (reference source - Clarke).
D. Confirmation by GCMS.

III. HEROIN

A. Spot tests.
B. GCMS preparation & confirmation.
C. IR preparation & confirmation.
D. Schedule.
E. References.

IV. METHAMPHETAMINE AND AMPHETAMINE

A. Spot tests.
B. GCMS preparation & confirmation.
C. IR preparation for D meth.
D. IR preparation for DL meth by PIT derivative.
E. Microcrystalline test.
F. Schedule.
G. References.

V. GENERAL UNKNOWNS

A. Spot tests.
B. GCMS preparation.
C. IR preparation.
D. References - Clarke and Mercke.

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VI. LSD

A. Screening test/TLC.
B. GCMS preparation.
C. GCMS conversion.
D. Schedule.
E. References.

VII. MUSHROOMS

A. Spot test.
B. TLC.
C. GCMS preparation.

VIII EXTRA TRAINING AREAS

A. FTIR tutorial.
B. GCMS tutorial.
C. Microcrystalline exercise.
D. Spot test exercise.
E. TLC exercise:
   1. T1 with caffeine/ephedrine/phenylpropanolamine and cocaine/heroin/meth mixed standards - UV, fluorescamine and iodoplatinate.
   2. T1 with LSD and mushrooms - UV & PDMAB.
   3. Acetone with LSD - UV & PDMAB.
F. Drug schedule DEA exercise.
G. Reading - Clarke and all photocopied material.

IX. PROFICIENCY TEST
At least 10 unknown samples - to include cocaine, heroin, methamphetamine, and non-controlled substances.

X. MOCK COURT

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Coeur d'Alene Drug Procedures

Spring 1998

General Unknown Sample by GC/MS

1. Dilute powder, solid or residue sample in MeOH or appropriate solvent.
2. Run on GC/MS and compare to computerized library match.
3. Run standard to confirm library match, look for similar retention times and ion scans.

General Unknown Sample by IR

1. Grind powder or solid sample with KBr.
2. Form pellet and run IR scan.
3. Compare scan with library match.

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COCAINE ANALYSIS:

I. Color test

COBALT thiocyanate=blue precipitate

Note: Free base cocaine may react slowly or not at all with cobalt thiocyanate. Free base may have to be converted to Cl -form- dissolve in hexane and bubble with concentrated HCl fumes; or add a drop of HCl to spot plate....with the presence of cocaine and cobalt thiocynate watch for color change to blue with precipitate.

II. Infrared spectrophotometer

1. Prepare KBr pellet---neat sample.
2. Run IR.
3. Compare to known standard.

III. GCMS

1. Extract sample with methanol.
2. Inject into GCMS. (HP Ultra 1 column,Temp.100-280, 20 degrees per minute, total run time 15 minutes.
3. Examine and compare retention times and ion scan to known standard.

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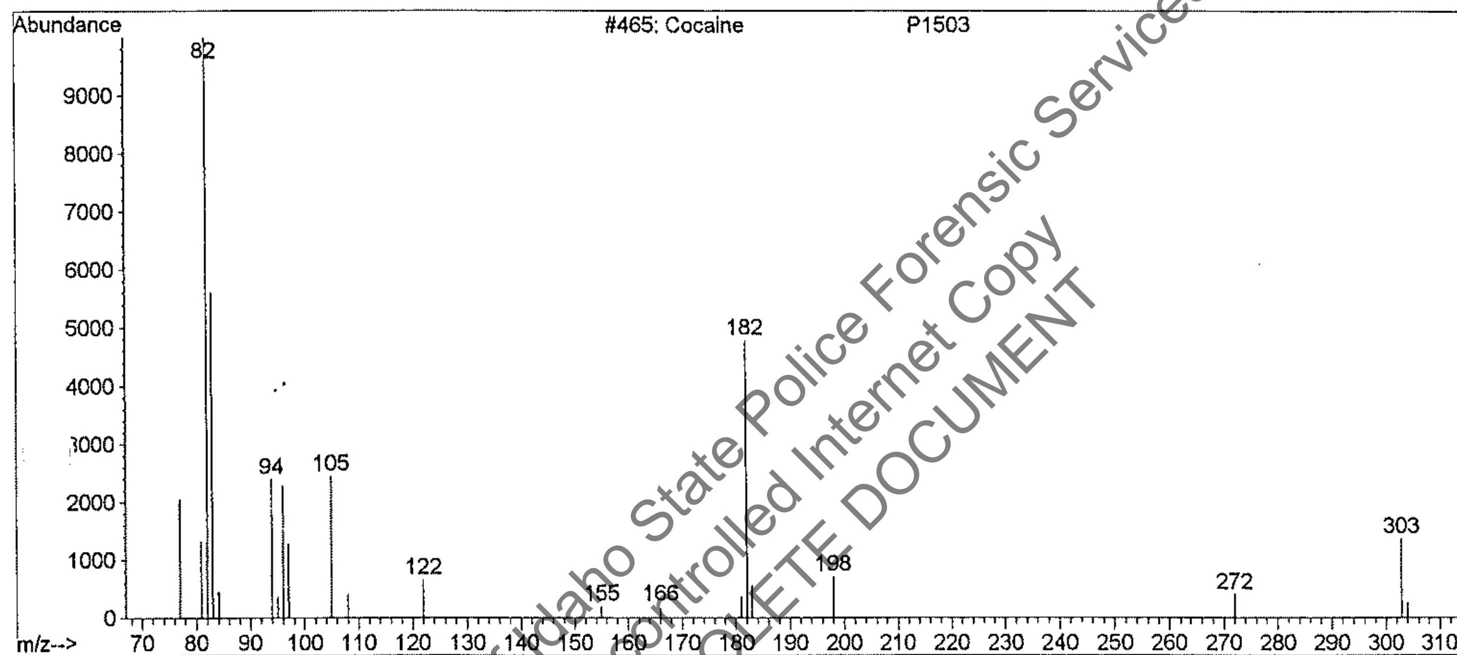
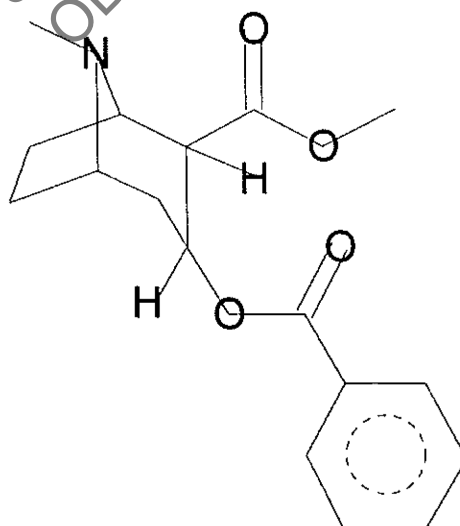
Cocaine P1503

Entry Number	465	from	D:\DATABASE\PMW_TOX2.L
CAS	000050-36-2		
Melting Point	0		
Boiling Point	0		
Retention Index	2200		
Mol Formula	C17H21NO4		
Mol Weight	303.147		
Company ID	Univ-Saar		

Miscellaneous Information
Local anesthetic Addictive drug %465





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Lysergic Acid Diethylamide (LSD) Analysis by GC/MS:

1. Immerse blotter acid in minimal amount of concentrated NH4OH for at least 10 minutes. (Blotter acid amount is 2 or 3 6mm X 6mm squares of sample)
2. Transfer solution into a 1.5 conical centrifuge tube.
3. Add 200ul of methylene chloride or diethyl ether.
4. Vortex.
5. Transfer extract into a micro-insert.
6. Run TLC plate (acetone for the solvent, visualize with pDMAB, light purple=positive)
7. Evaporate
8. Add 30-200ul of MSTFA.
9. Analyze for TMS derivative on GC/MS.

GC/MS recommendation: Column 20m Ultra 1 HP, Temperature: 100-315 degrees (15 minute hold) at 10 degrees per minute.

CONFIRMATION OF POSITIVE:
Match of TMS derivative spectra to known standard.

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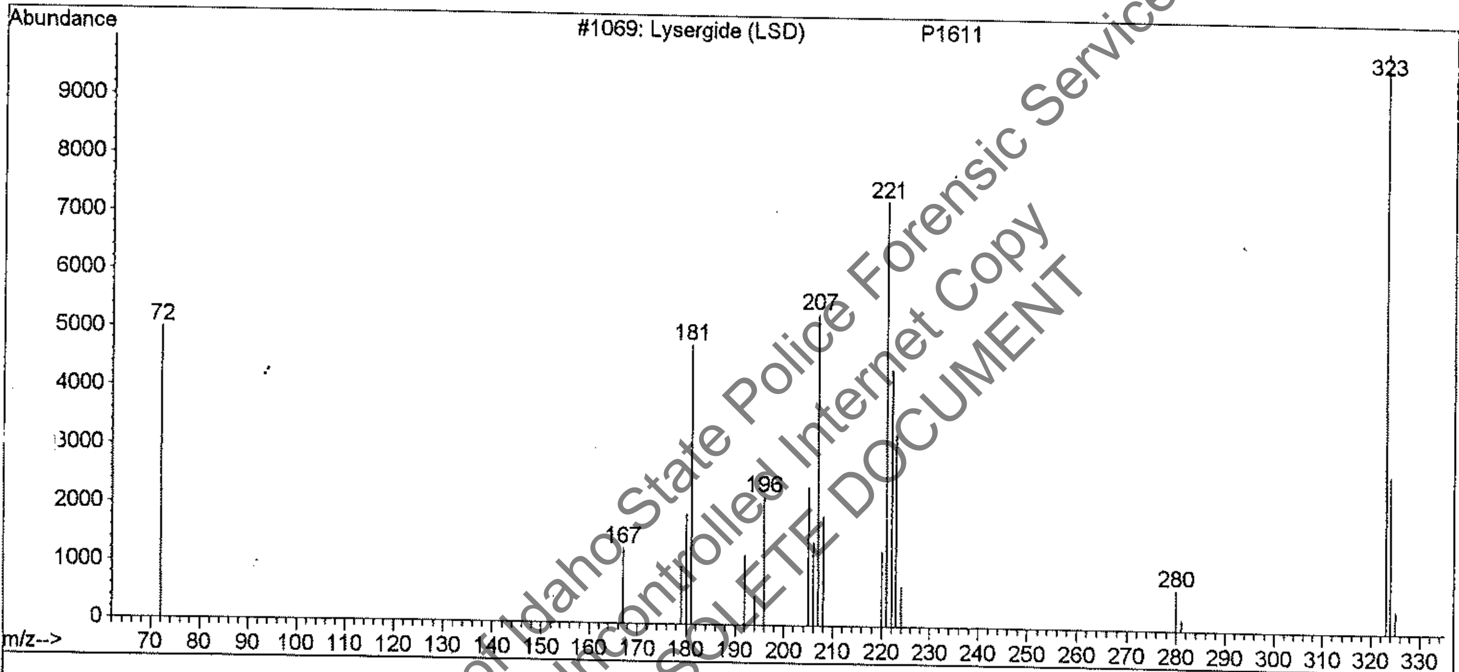
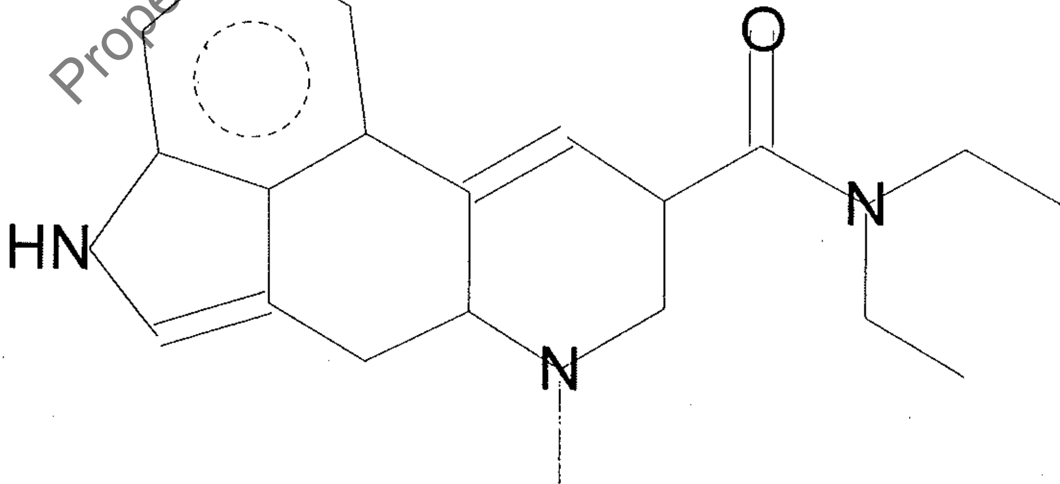
Lysergide (LSD) P1611

Entry Number 1069 from D:\DATABASE\PMW_TOX2.L
CAS 000050-37-3
Melting Point 0
Boiling Point 0
Retention Index 3445
Mol Formula C20H25N3O
Mol Weight 323.199
Company ID Univ-Saar

Miscellaneous Information
Psychedelic %1069





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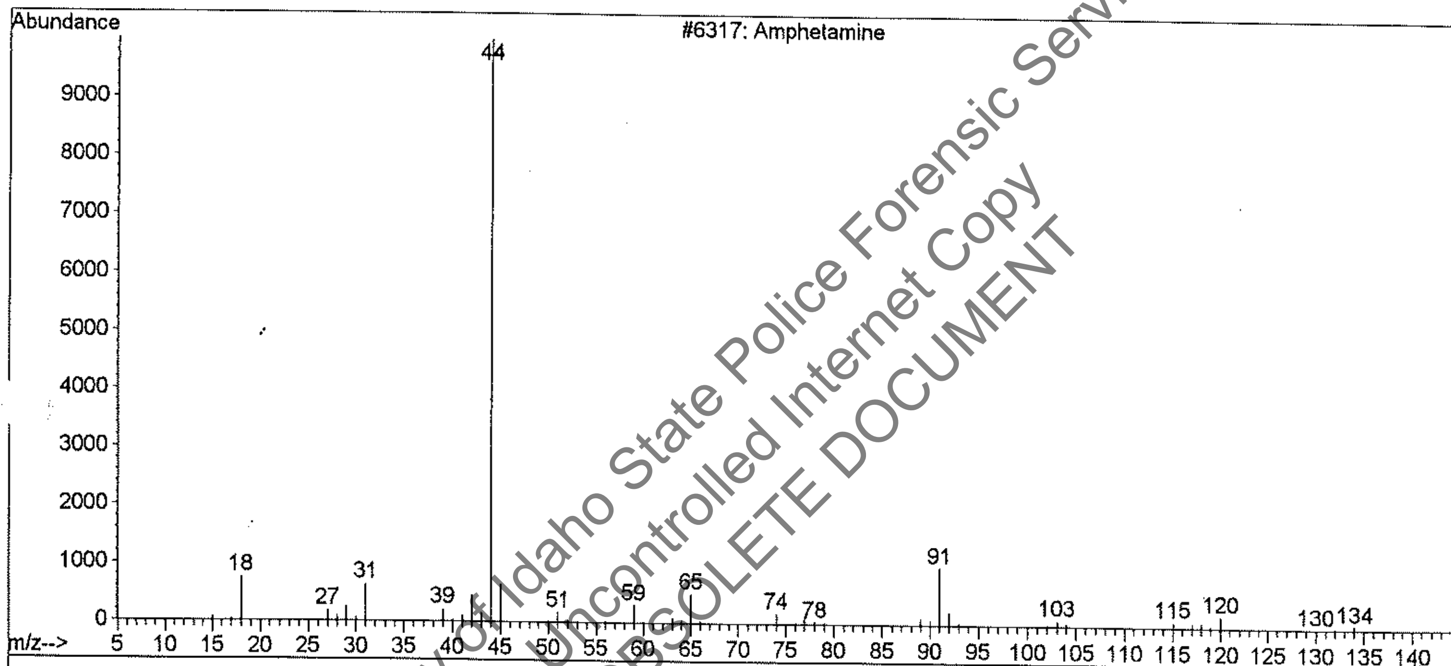
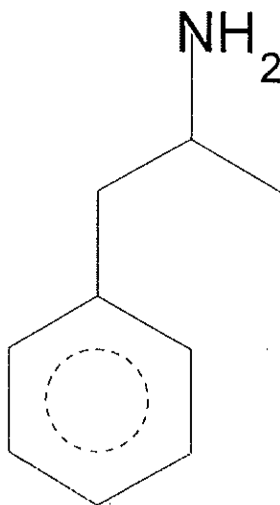
Amphetamine

Entry Number 6317 from D:\DATABASE\NBS75K.L
CAS 000300-62-9
Melting Point -300
Boiling Point -300
Retention Index 0
Mol Formula C9H13N
Mol Weight 135
Company ID NIST 1992

Miscellaneous Information
QI=64 Steroids; Alkaloids; Drugs; Derivatives; Metals; Misc
. Natural products; Fatty acids and Lipids;





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METHAMPHETAMINE/AMPHETAMINE ANALYSIS:

1. Add .5N NaOH (2.5ml of 2N to 10ml of DI water) to a small amount of sample powder.
2. Extract with hexane (1 to 3 times ....depending on the purity of the sample)
3. Wash with H2O (1 to 3 times as necessary).
4. Dry hexane thru NaSO4.
5. Bubble with HCl fumes until precipitate forms (may use light heat for recrystallizing).
6. Filter crystals and allow to dry.
7. Run IR and compare to known standard. If poor IR is obtained, run on GC/MS. GC/MS recommendations: HP Ultra 1 Column 20m, 100-280°C, 20°C/minute, inlet temp. 175°C.

TOXI-LAB PROCEDURE:

1. Place a blank toxilab disc into a metal concentration cup.
2. Add a few drops of sample extract to the paper disc in the metal cup.
3. Allow the disc to dry with a gentile current of warm air.
5. Place the disc in to the Toxigram at the appropriate opening.
6. Allow the "loaded" Toxi-gram to warm on a hot plate for a few seconds face up.
7. Place 3ml of amine solution and 20ul of ammonium hydroxide in to a chromatography chamber and swirl vigorously for 10 seconds.
8. Remove the Tox-gram from the warmer and lower disc end first into the chromatography chamber. Do not allow the sides of the Toxi-gram to tough the walls of the chamber.
9. Remove the Toxi-gram when the dye spots on the Toxi-gram reach 9.5cm (after 12-17 minutes) and place the Toxi-gram face down on the warmer for 30-60 seconds or until the fumes have evaporated.
10. Place the Toxi-gram into a jar (A-1) that contains formaldehyde under a plastic screen which allows only the fumes to reach the Toxi-gram. Leave in the jar for 2-15 minutes.
11. Allow the Toxi-gram to warm for a few seconds on the warmer.
12. Dip the Toxi-gram slowly into concentrated sulfuric acid (A-2). Remove and note the colors.
13. Dip the Toxi-gram slowly into H20. Remove and note the colors.
14. Place the Toxi-gram under UV light, note the colors.
15. Dip the Toxi-gram unto modified dragendorff's solution, note the colors.
16. Place the Taxi-gram between two sheets of glass, make a photo copy and keep with the report.

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CONFIRMATION OF POSITIVE:

Must have IR match to a known standard .

OR

Must have GC/MS spectra match and similar retention times to a known standard and Toxilab confirmation.

Color test positives: Amphetamine............Marquis=Orange
Methamphetamine.....Marquis=Orange
.....Secondary Amine=Inky Blue

Methamphetamine/Amphetamine Acetylation Method.

1. Dissolve small amount of sample in H2O.
2. Make very basic with NaOH.
3. Extract with Petroleum Ether or Hexane.
4. Split sample in half and place in autoinjection vials.
5. Acetylate one half of the sample with 1-2 drops of acetic anhydride.
6. Run on the GC/MS; compare retention time and ion scans to known standards. GC/MS recommendations: HP Ultra 1 20m column, Temperature 100-280°C, 20°C/minute, total run time 15 minutes.

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Methamphetamine Hydrochloride

Entry Number	9523	from	D:\DATABASE\NBS75K.L
CAS	000537-46-2		
Melting Point	-300		
Boiling Point	-300		
Retention Index	0		
Mol Formula	C10H15N		
Mol Weight	149.12		
Company ID	NIST 1992		

Miscellaneous Information
QI=65 Steroids; Drugs; Amino Acids; Metals; Carbohydrates;
Misc. Natural products; Fatty acids and Lipids;

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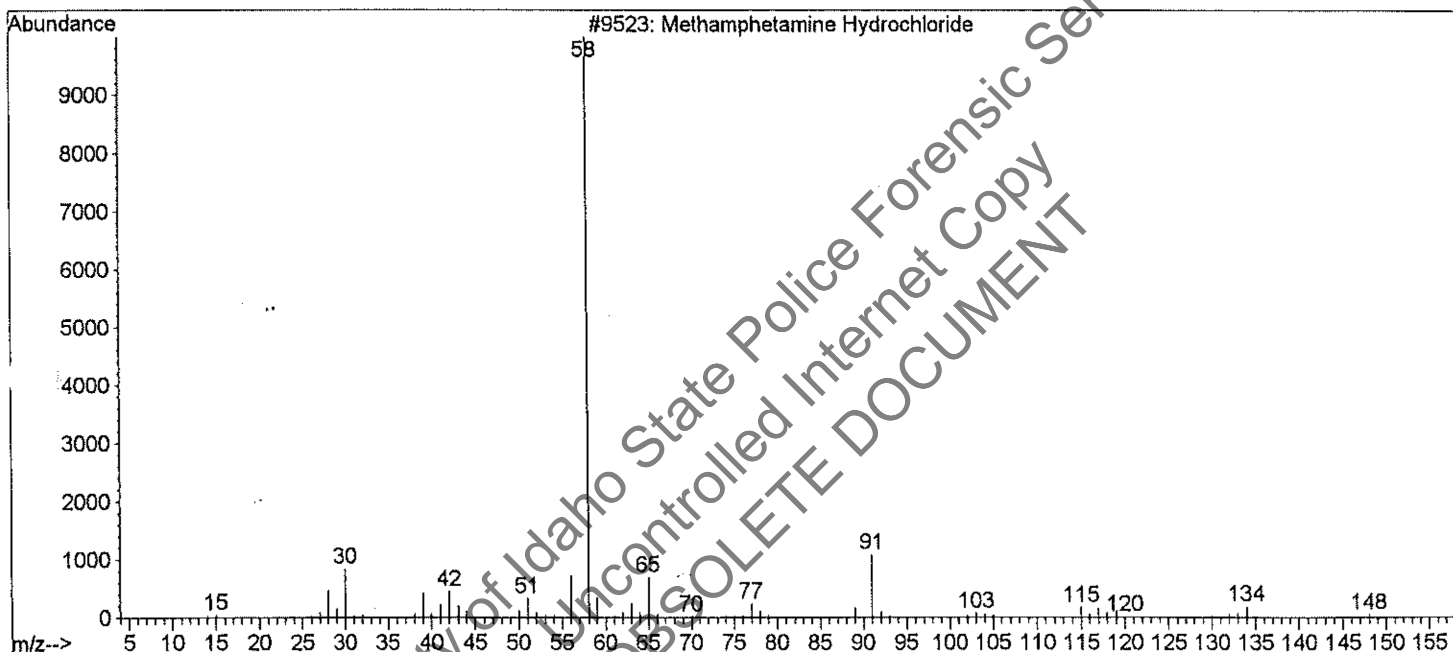

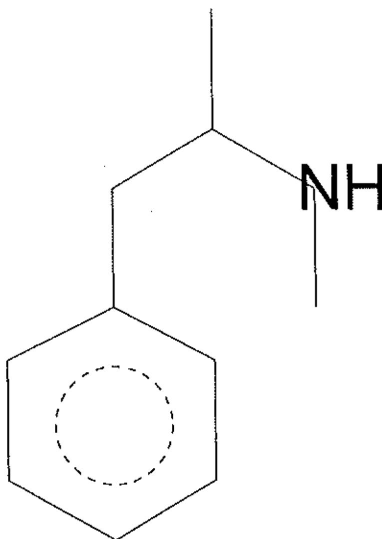

MESCALINE ANALYSIS:

1. Dissolve in .1N H_2SO_4.
2. Wash with $CHCl_3$.
3. Make basic (pH 8) with Na_2CO_3.
4. Extract into CHCL3
5. Filter and evaporate.
6. Run IR and compare to known standard.
7. GC/MS recommendation: Column: HP 20m Ultra 1, Temperature: 100-280 degrees 20 degrees per minute for 30 minute total run time. Inlet temp: 175 degrees C. Compare retention time and ion scans to known standard.

Color test:
Marquis: orange
Mecke's: orange turning brown
Libermann's: black
Con. HNO_3: fast dark red

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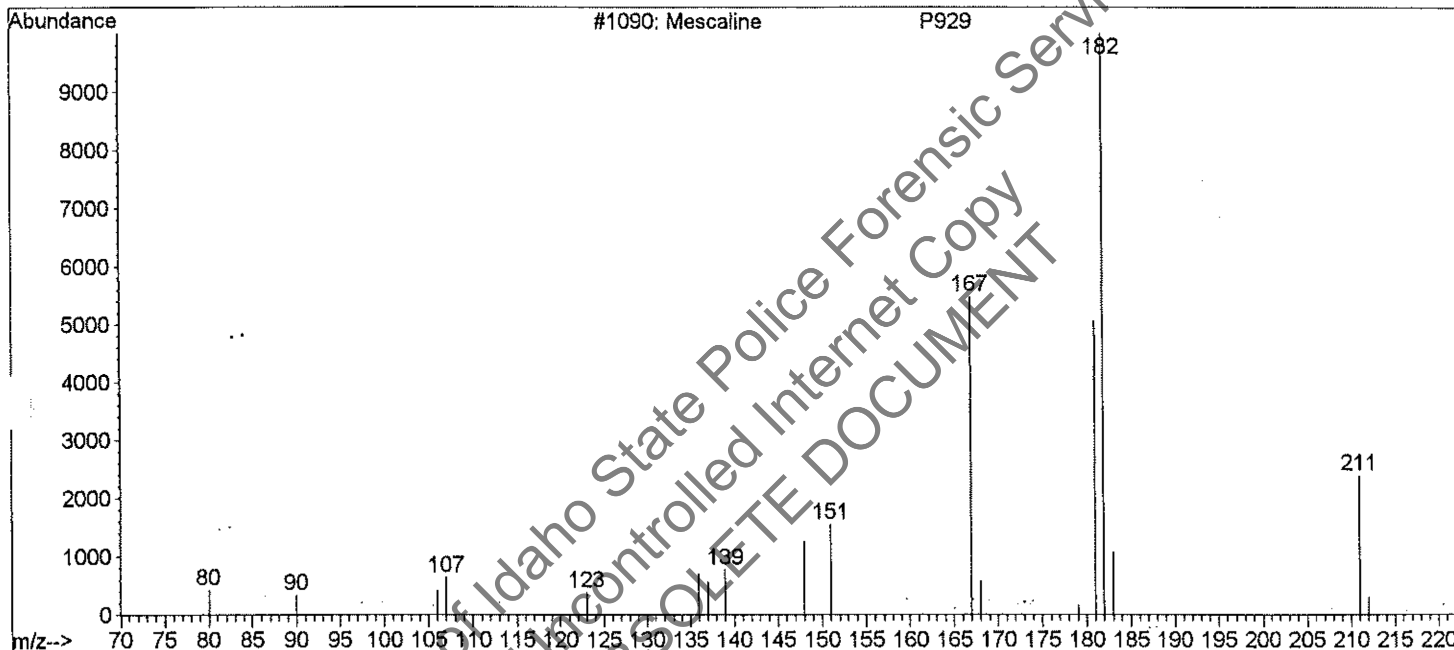
Mescaline P929

Entry Number	1090	from	D:\DATABASE\PMW_TOX2.L
CAS	000054-04-6		
Melting Point	0		
Boiling Point	0		
Retention Index	1690		
Mol Formula	C11H17NO3		
Mol Weight	211.12		
Company ID	Univ-Saar		

Miscellaneous Information
Psychedelic %1090



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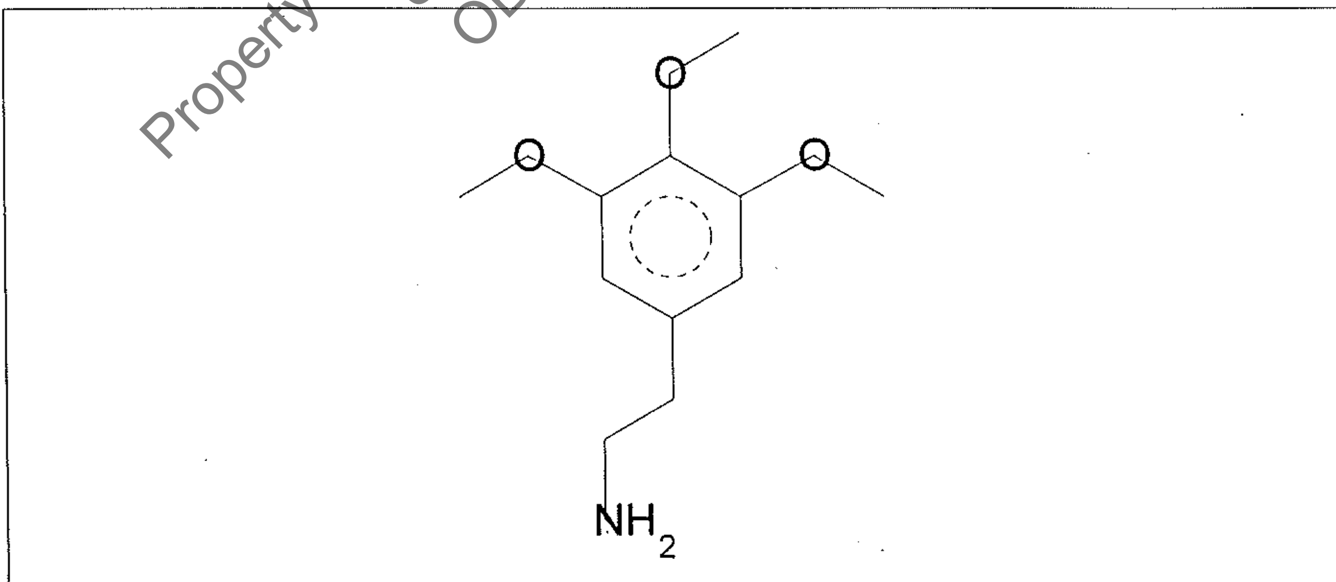

Mushroom Extraction:

1. Allow mushrooms to dry if needed..
2. Grind @ 0.5 -3.0 grams of mushrooms to a fine powder.
3. Place powder in a 12.5 cm filter paper folder to fit a glass funnel.
4. Rinse mushroom powder with approx. 50ml of chloroform.
5. Discard the CHCl3
6. Allow mushroom powder to dry completely.
7. Rinse mushroom powder with approx. 50ml of 0.1N sulfuric acid COLLECT rinsings in a centrifuge tube.
8. Transfer half the extract in to another centrifuge tube (now have 25ml in two 50ml tubes)
9. Add equal volumes of CHCl3 to each of the tubes and wash (mix with lid by hand)
10. Centrifuge for about 2 minutes.
11. Remove the aqueous layers an place into two new centrifuge tubes.
12. Make the aqueous solutions basic with 1N NaOH *****Check the pH.
13. Add to the basic aqueous solutions equal volumes of methylene chlorine (Ch2Cl2).
14. Mix by hand and centrifuge for 2 minutes.
15. Aspirate the aqueous. (No need to keep, bottom layer is now of importance).
16. Transfer the extract (bottom layer) in to a 50ml beaker and boil to about 1-2ml. Do not go to dryness.
17. Transfer extract in to autoinjection vial for GC/MS. GC/MS recommendations: HP ultra 1 Column, 100-280°C, 20°/ minute, total run time 30 minutes.

For TLC plate :

18. Allow the 50 ml beaker to dry completely.
19. Reconstitute the beaker with a few drops of methanol.
20. Spot the sample directly from the beaker on to a TLC plate.
21. Run with a blank and the standard in a T1 solvent.
    T1 Solvent: 7.5 drops NH4OH and 25ml of MeOH.
    Visualize with: 1. Fast Blue BB——Red Spot
    2. p-DMAB———Blue Spot

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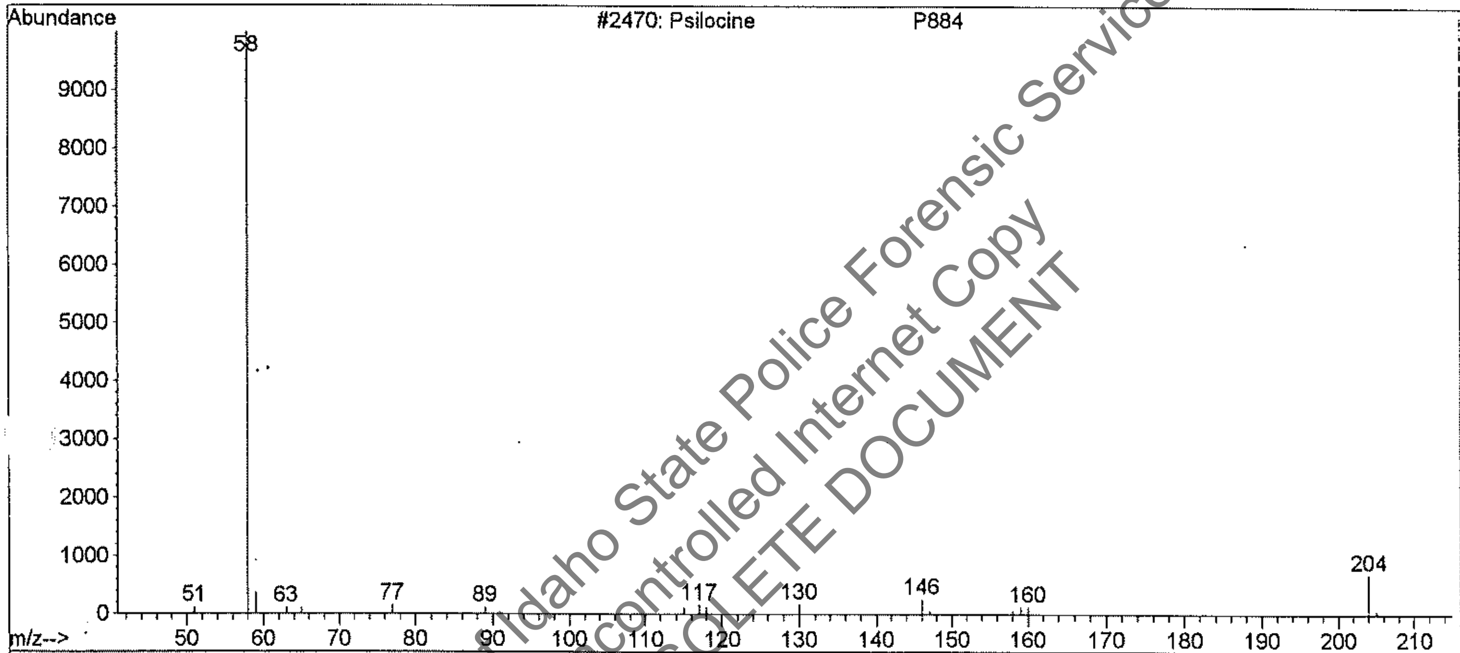
Confirmation of Positive for Psilocin:

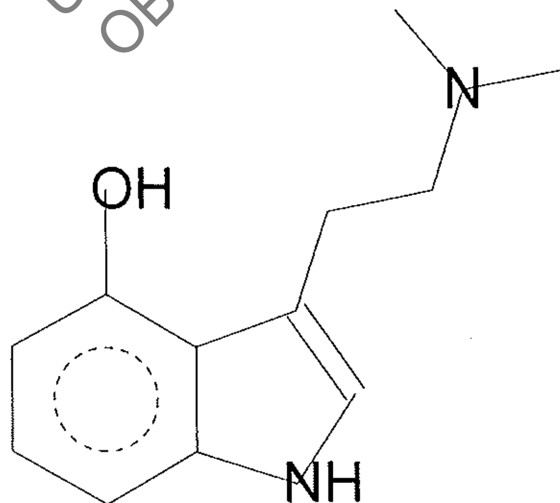
1. GCMS spectra match to psilocin standard run on the same day, along with a blank of the same solvent.
2. TLC plate with match to Rf location to known standard.

Psilocine P884

Entry Number 2470 from D:\DATABASE\PMW_TOX2.L
CAS 000520-53-6
Melting Point 0
Boiling Point 0
Retention Index 1995
Mol Formula C12H16N2O
Mol Weight 204.126
Company ID Univ-Saar

Miscellaneous Information
Psychedelic %2470





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MARIJUANA ANALYSIS:

I. General Testing Procedure

A. Microscopic Examination
Plant material must possess the following characteristics:

1. CYSTOLITHIC HAIRS
Small claw like protrusions along the topside and edges of the leaf.
2. FINE HAIRS
Long tapered hairs on the underside of the leaf, which give a fuzzy appearance.

Seeds must possess the following characteristics:

1. MOSAIC PATTERNED SHELL
Veining gives the shell a mosaic appearance.
2. TORTOISE SHELL-LIKE SHAPE
Shell resembles a small coconut with a ridge that runs along its circumference.

B. Chemical Test--Modified Duquenois Levine
This test independently supports the microscopic examination.

1. Pet ether is added to actual plant material in a small test tube.
2. Decant a portion of pet ether into an additional test tube.
3. Evaporated the pet ether.
4. Add a few drops of the Duquenois reagent to the extract.
5. Add an equal amount of concentrated hydrochloric acid to the test tube and mix.

***If marijuana is present, a deep purple colored complex will result. The purple color will form if any phenolic compound with an alkyl side chain is present, e.g. resorcinol.

6. Add a small amount of chloroform to the test tube.

***If the purple complex enters the chloroform (that is it is soluble in CHCl3), the entire test is POSITIVE for the presence of Marijuana. This is a crucial part of the test sequence. If the purple color does not enter the chloroform layer, the test is considered NEGATIVE.

C. Thin Layer Chromatography (TLC)

1. The pet ether remaining in the test tube with actual plant material is spotted on to two appropriately labeled silica gel thin layer chromatography plates.
2. A marijuana standard and a blank of pet ether is also spotted on the plates.
3. Then the plates are placed into ONE of the following solvent systems:
    a. Hexane-diethlyether 4:1
    b. Benzene
    c. Chloroform
    d. Petroleum ether-methanol. 95:5 (use if suspect PCP)
    (Two systems must be used if no microscopic tests was done.)
4. Plates are visualized by spraying with a Fast Blue BB salt solution.
Separated cannabinoid components of extract will form a characteristic color:

e.g. Hexane/diethly ehter 4:1
Cannabidiol (CBD)------Orange

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Tetrahydrocannabidinol (THC)-----Scarlet
Cannabinol (CBN)-----Purple
5. Visualized spots are compared to the marijuana standard. If scarlet spot for THC is present, the test is considered POSITIVE.

A. Germination of Seeds (Done to prove manufacturing)
1. Place seeds in moist container.
2. Seeds should germinate in two the three days.
3. Seeds are considered fertile if more than 10% sprout.
4. Estimate what percentage sprouted.

II. Requirements for Specific Samples:

A. Plant material.
1. Microscopic.
2. Modified Duquenois Levine
3. TLC

B. Seeds
1. Microscopic
2. Modified Duquenois Levine
3. TLC
4. Germination

C. Hash
1. Microscopic (optional) Dissolve in $CHCl_3$ and look for crushed plant hairs.
2. Modified Duquenois Levine
3. Two different TLC systems.

D. Pipes
1. Microscopic on ash (optional)
2. Modified Duquenois Levine\
3. Two different TLC systems----only one TLC system is required if microscopic is positive.

III. CONFIRMATION OF POSITIVE:

The sample may be designated as "marijuana, non-narcotic, schedule 1" if microscopic, Duquenois and TLC are all positive.

The sample may be designated as "the resins of marijuana, non-narcotic, schedule 1" if microscopic was not done, but the Duquenois and two TLC systems were positive.

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BARBITURATES

1. Solubility:
    a. Sodium salts -- generally soluble in H_2O
    b. Free acid -- soluble in ethanol, chloroform and ether

2. Color Test:
    a. Dilli-Koppanyi -- violet
    b. Zwikkers -- formation of a colored barbiturate-$CuSO_4$ complex.
    c. Libermanns

3. Crystal Tests:
    a. $KOH\backslash H_3POH_4$ free acid
    b. H_2SO_4
    c. Wagenaars ($CuSO_4$)
    d. Davis ($AgNO_3$)

4. Sample Preparation:
    a. Most barbiturates, whether encountered as the sodium salt or as the free acid, can be extracted from dilute Hcl into ether. Particle picking the contents of capsules is often more efficient than extracting.
    b.
        - dissolve sample in KOH
        - wash with methylene chloride
        - acidify with concentrated H_2SO_4, and extract into ether
    c. - dissolve in 9:1 chloroform/methanol

5. GCMS
    a. 100°C to 280°C at 20°/minute ramp, 4 min. solvent delay
    30 meter, HP Ultra II column
    1 ul injection

    MS: Most barbiturates can be distinguished by their mass spectra, however closely related isomers may be difficult to identify.

6. IR: Barbiturates are difficult to differentiate by IR unless scanned down to 250 cm^{-1}. The region between 700 and 250 cm^{-1} contains the characteristic data for the functional groups of the barbiturate. Spectra variation can occur depending on how the sample is prepared, although the far IR region is less susceptible to variation.

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COCAINE (methylbenzoylecgonine)

1. Solubility:
   a. Free Base -- 1 to 0.5 $CHCl_3$, 1 to 4 ether, 1 to 600 H_2O
   b. HCl salt -- 1 to 0.5 H_2O, 1 to 4 EtOH, ether insoluble

2. Color Test:
   a. Ruybal -- Blue
   b. Cobalt thio -- Blue ppt.

3. Crystal Tests:
   a. 5% $AuCl_3$/HOAc -- T.V. antennas, stars, serrated needles
   b. $PtCl_4$/H_2O -- Molars

4. TLC:
   a. solvent -- $CHCl_3$:MeOH (4:1)
      location -- Acidified iodoplatinate spray

5. Sample Preparation:
   a. - dissolve sample in acidic H_2O and filter.
      - make basic with saturated aqueous $NaHCO_3$, cocaine base will precipitate out of solution.
      - extract cocaine base with petroleum ether and allow to evaporate and crystallize on a watch glass.
   b. - dissolve aliquot in 9:1 chloroform/methanol

6. GCMS:
   a. 100°C - 280°C at 20°C/min. ramp, 4 min. solvent delay
      30 meter HP Ultra II column, 1 ul injection

      MS:
        principal peaks at m/z 82, 182, 83, 105, 303, 77, 94, 96.

7. IR:
   a. principal peaks at wavenumbers 1710, 1738, 1275, 1110, 712, 1037

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DIAZEPAM

1. Solubility:
   a. 1 to 2 in $CHCl_3$, 1 to 25 in ethanol, 1 to 39 in ether, slightly soluble in H_2O

2. TLC:
   a. solvent -- Methanol:concentrated NH_4OH, 100:1.5
      location -- Acidified iodoplatinate spray

3. Sample Preparation:
   a. - tablets may be ground and dry extracted with $CHCl_3$ or CH_2Cl_2.

4. GCMS:
   a. - 100°C to 280°C at 20°C/minute ramp, 4 min. solvent delay
      30 meter HP Ultra II column, 1 ul injection

   MS:
      principal peaks at m/z 256, 283, 284, 285, 257, 255, 258, 286.

5. IR:
   a. principal peaks at wavenumbers 1681, 1313, 706, 840, 1125, 740.

   Note: This compound often forms a glass. Grinding the sample with a small quantity of pentane or petroleum ether will usually give a more crystalline sample.

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EPHEDRINE (PHENYLMETHYLAMINOPROPANOL) and/or PSEUDOEPHEDRINE

1. Solubility:
   a. Free Base -- 1 to 20 H_2O, 1 to <1 EtOH, ether, $CHCl_3$ with turbidity due to separation of water.
   b. HCl salt -- 1 to 3 or 1 to 4 H_2O, 1 to 14 or 1 to 17 EtOH, slightly soluble in $CHCl_3$, practically insoluble in ether.

2. Color Test:
   a. Liebermann -- Bright yellow

3. Crystal Tests:
   a. $AuCl_3/H_3PO_4$ without 520nm plate -- alligator skin. long jagged plates

4. TLC:
   a. solvent -- $CHCl_3$:MeOH (4:1)
      location -- Acidified iodoplatinate spray, brown/black spot.

5. Sample Preparation:
   a. - dissolve sample in H_2O and filter.
      - wash with $CHCl_3$. Save H_2O layer.
      - make basic with saturated NaOH, ephedrine base will precipitate out of solution.
      - extract with $CHCl_3$ (petroleum ether will also work) and acidify with acidic ether. Ephedrine HCl will crystalize on evaporation on a watch glass.
   b. - dissolve aliquot in 9:1 chloroform/methanol

6. GCMS:
   a. 100°C to 280°C at 20°C/minute ramp, 4 min. solvent delay 30 meter HP Ultra II column, 1 ul injection

   MS:
   principal peaks at m/z 58, 77, 56, 51, 79, 59, 105, 91, 131, 117, 119, 146, 148.

7. IR:
   a. principal peaks at wavenumbers 1590, 1460, 1047, 991, 752, 702.

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HEROIN (diacetylmorphine)

1. Solubility:
   a. Base -- 1 to 1.5 $CHCl_3$, 1 to 100 ether, 1 to 1700 H_2O
   b. HCl salt -- 1 to 1.6 H_2O, 1 to 12 EtOH, ether insoluble

2. Color Test:
   a. Marquis -- violet
   b. Mecke -- olive-green, grey
   c. Frödhe -- violet, mauve

3. TLC:
   a. solvent -- $CHCl_3$:MeOH (4:1)
      location -- Acidified iodoplatinate spray

4. Sample Preparation:
   a. - dissolve sample in acidic H_2O and filter.
      - extract with $CHCl_3$, retain organic layer.
      - extract with neutral H_2O, retain aqueous layer.
      - make basic with saturated aqueous $NaHCO_3$, heroin base will precipitate out of solution.
      - extract heroin with $CHCl_3$, and allow to evaporate.
      - crystallize in $CHCl_3$:Heptane (1:3).
   b. - dissolve aliquot in 9:1 chloroform/methanol

5. GCMS:
   a. 100°C to 280°C at 20°C/min. ramp. 4 min. solvent delay
      30 meter HP Ultra II column, 1 ul injection

   MS:
      principal peaks at m/z 327, 43, 369, 268, 310, 42, 215

6. IR:
   a. principal peaks at wavenumbers 1245, 1764, 1178, 1215, 911, 1736.

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LYSERGIC ACID DIETHYLAMIDE (LSD)

1. Solubility:
   a. Base -- soluble in alcohol, chloroform, ether.
   b. Tartrate -- soluble in water, dilute acids.

2. Color Test:
   a. Paper squares treated with LSD will generally give an overall blue-white glow under long wave UV light.
   b. Ehrlichs -- purple.

3. TLC:
   a. solvent systems -- acetone, chloroform:acetone 1:4
      procedure -- Spot sample vs. known LAMPA and LSD. Irradiate with long wave UV for about 3-4 min. (spots should glow blue-white under uv and turn tan without uv). Place in solvent tanks.
      After drying plates, circle with pencil the LSD (visible as lavender spots under uv longwave light). Overspray with Ehrlichs and the spots will turn purple.

4. Sample Preparation:
   a. - place sample into a small centrifuge tube and cover with concentrated NH_4OH.
      - let sit for 5-10 minutes, remove paper from liquid.
      - add 200 ul chloroform, mix and centrifuge.
      - put organic layer into micro-insert and dry (no heat).
      - add 200 ul MSTFA

5. GCMS:
   a. 280°C isothermal, 26 minute solvent delay
      30 meter HP Ultra II column, 2 ul injection

   MS:
      principal peaks at 323, 221, 181, 222, 207, 72, 223, 324
      (LSD has 29 ion and small 43 ion to distinguish from LAMPA)

6. IR:
   a. principal peaks at wavenumbers 1626, 1307, 1136, 1066, 1212, 749.

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MARIJUANA (Cannabis sativa)

Marijuana usually refers to the green vegetable material comprised of leaves and flowering tops of the plant Cannabis sativa. Hashish usually refers to the resins extracted from the plant material. The mature plant contains a number of psychoactive components collectively known as cannabinoids.

1. Solubility:
   a. Cannabinoids -- soluble in $CHCl_3$, alcohols, acetone, and petroleum ether. Practically insoluble in H_2O.

2. Color Test:
   a. Duquenois-Levine -- blue-indigo, extracts into $CHCl_3$.

3. Morphological characteristics:
   a. The vegetable material has simple and cystolithic hairs on opposite sides of the same leaf fragment.
   b. Seeds have veins and resemble a coconut.

4. TLC:
   a. solvent -- hexanes/ether (4:1)
      -- toluene or benzene
      location -- Fast Blue BB

5. Sample Preparation:
   a. - Dry extract with petroleum ether.

6. GCMS:
   a. 250°C isothermal
   MS:
   principal peaks at m/z
   delta-9-THC -- 299, 231, 314, 43, 41, 295, 55, 271
   delta-8-THC -- 221, 314, 248, 261, 193, 236, 222, 315
   cannabidiol -- 231, 246, 314, 232, 121, 193, 74, 174
   cannabinol -- 295, 296, 238, 310, 119, 43, 251, 239

7. IR:
   a. principal peaks at wavenumbers:
   delta-9-THC -- 1580, 1030, 1620, 1180, 1130, 1050
   delta-8-thc -- 1580, 1040, 1620, 1180, 1080, 1260
   cannabidiol -- 1585, 1630, 1020, 1210, 1240, 1050
   cannabinol -- 1620, 1050, 1580, 1030, 1120, 1228

Note: Marijuana requires a microscopic examination followed by Duquenois-Levine color test. The identification requires a training program not presented in this manual. See training manual.

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METHADONE

1. Solubility:
   a. Free Base -- soluble in organic solvents
   b. HCl salt -- 1 to 12 H_2O, 1 to 7 EtOH, ether insoluble

2. Color Test:
   a. Liebermann -- Brown-orange

3. TLC:
   a. solvent -- Methanol:concentrated NH_4OH, 100:1.5
      location -- Acidified iodoplatinate spray

4. Sample Preparation:
   a. - this compound forms ion pairs.
      - dissolve the sample in 3N HCl.
      - extract the HCl salt with $CHCl_3$ or CH_2Cl_2.

   b. - the base extracts from alkaline aqueous solutions into
a   suitable organic solvent, eg. CH_2Cl_2.

5. GCMS:
   a. 250°C isothermal

   MS:
   principal peaks at m/z 72, 73, 91, 293, 223, 165, 85, 71

6. IR:
   a. principal peaks at wavenumbers 1709, 710, 1107, 769, 1107,943 and 1133.

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METHAMPHETAMINE

1. Solubility:
   a. Free Base -- soluble in H_2O, miscible with EtOH, $CHCl_3$, and ether.
   b. HCl salt -- 1 to 2 H_2O, 1 to 4 EtOH, 1 to 5 $CHCl_3$, practically insoluble in ether.

2. Color Test:
   a. Marquis -- rapid bright orange/brown.
   b. Secondary amines -- rapid blue

3. Crystal Tests:
   a. $AuCl_3/H_3PO_4$ without 520nm plate -- calipers.

4. TLC:
   a. solvent -- $CHCl_3$:MeOH (4:1)
      location -- Acidified iodoplatinate spray - blue spot

5. Sample Preparation:
   a. - dissolve sample in dry $CHCl_3$, filter and dry down.
      - dissolve in acidic H_2O, wash with ether and save H_2O layer.
      - make basic with saturated NaOH, base form of methamphetamine will precipitate out of solution.
      - extract with $CHCl_3$, add acidic ether to precipitate acid form of methamphetamine. Allow to dry.
   b. - dissolve aliquot in 9:1 chloroform/methanol.

6. GCMS:
   a. 100°C to 280°C at 20°/min. ramp, 4 min. solvent delay
      30 meter HP Ultra II column, 1 ul injection
   MS:
      principal peaks at m/z 58, 91, 65, 56, 134, 59, 77, 115, 117, 119, 89, 42, 148.

7. IR:
   a. principal peaks at wavenumbers 1491, 1456, series of 7 peaks centered at 1060, 752, 702.

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PSILOCYN

The psychoactive ingredient most commonly seen in the mushroom genus Psilocybe.

1. Solubility:
   a. Soluble in ethanol and dilute acid.

2. Color Test:
   a. Marquis -- green-brown
   b. Fast Blue B -- red, add conc. HCl -- blue

3. TLC:
   a. solvent -- T1 [MeOH:NH_4OH (20:1)]
      location -- Fast Blue BB (red), then conc. HCl (blue)

4. Sample Preparation:
   a. - grind mushroom material and soak in 0.1N HCl for 30 min.
      - filter and wash twice with CH_2Cl_2 using a separatory funnel. Discard the CH_2Cl_2.
      - make the solution basic with $NaHCO_3$.
      - extract with CH_2Cl_2 and concentrate the solution.

5. GCMS:
   a. 100 - 280°C, 20°C/min. ramp, 4 min. solvent delay
      30 meter HP Ultra II column, 1 ul injection
   MS:
      principal peaks at m/z 58, 204, 59, 42, 30, 146, 77, 44

6. IR:
   a. principal peaks at wavenumbers 836, 1261, 1236, 1042, 1061, 733.

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REGULATED STEROIDS

Encountered as tablets(2.5 to 50 mg), aqueous suspensions and injectable oils (10 to 100 mg/ml). Labels are not reliable -- over half of encountered samples have been counterfeit.

1. Solubility:
   a. Insoluble in H_2O. Soluble in alcohols, ether, most organic solvents.

2. Color Test:
   a. 2-Naphthol/H_2SO_4
   b. H_2SO_4/longwave uv

3. Sample Preparation:
   a. tablets
      - dry extract with methanol, ether or CH_2Cl_2.

   b. oils
      - extract with 9:1 methanol/water.

   c. aqueous suspensions
      - add methanol

   Caution: Oils may cause GC column problems - try Sep/Pak columns for clean-up. Enols are not stable in methanol - try silating with BSTFA.

4. GCMS:
   a. 250 to 280°C isothermal runs recommended.

   MS:
   scan up to 480 amu.

5. IR:
   a. polymorphism and solvation are frequently encountered. Epimers are easily distinguishable by IR. Esters are difficult to distinguish, differing often only by the length of side chains.

Also see DEA Steroid Workshop reference book.

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COCAINE ANALYSIS:

I. Color test

COBALT thiocyanate=blue precipitate

Note: Free base cocaine may react slowly or not at all with cobalt thiocyanate. Free base may have to be converted to Cl -form- dissolve in hexane and bubble with concentrated HCl fumes; or add a drop of HCl to spot plate....with the presence of cocaine and cobalt thiocynate watch for color change to blue with precipitate.

II. Infrared spectrophotometer

1. Prepare KBr pellet---neat sample.
2. Run IR.
3. Compare to known standard.

III. GCMS

1. Extract sample with methanol.
2. Inject into GCMS. (HP Ultra 1 column,Temp.100-280, 20 degrees per minute, total run time 15 minutes.
3. Examine and compare retention times and ion scan to known standard.

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Lysergic Acid Diethylamide (LSD) Analysis by GC/MS:

1. Immerse blotter acid in minimal amount of concentrated NH_4OH for at least 10 minutes. (Blotter acid amount is 2 or 3 6mm X 6mm squares of sample)
2. Transfer solution into a 1.5 conical centrifuge tube.
3. Add 200ul of methylene chloride or diethyl ether.
4. Vortex.
5. Transfer extract into a micro-insert.
6. Run TLC plate (acetone for the solvent, visualize with pDMAB, light purple=positive)
7. Evaporate
8. Add 30-200ul of MSTFA.
9. Analyze for TMS derivative on GC/MS.

GC/MS recommendation: Column 20m Ultra 1 HP, Temperature: 100-315 degrees (15 minute hold) at 10 degrees per minute.

CONFIRMATION OF POSITIVE:
Match of TMS derivative spectra to known standard.

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MARIJUANA ANALYSIS:

I. General Testing Procedure

A. Microscopic Examination

Plant material must possess the following characteristics:

1. CYSTOLITHIC HAIRS

Small claw like protrusions along the topside and edges of the leaf.

2. FINE HAIRS

Long tapered hairs on the underside of the leaf, which give a fuzzy appearance.

Seeds must possess the following characteristics:

1. MOSAIC PATTERNED SHELL

Veining gives the shell a mosaic appearance.

2. TORTOISE SHELL-LIKE SHAPE

Shell resembles a small coconut with a ridge that runs along its circumference.

B. Chemical Test--Modified Duquenois Levine

This test independently supports the microscopic examination.

1. Pet ether is added to actual plant material in a small test tube.
2. Decant a portion of pet ether into an additional test tube.
3. Evaporated the pet ether.
4. Add a few drops of the Duquenois reagent to the extract.
5. Add an equal amount of concentrated hydrochloric acid to the test tube and mix.

***If marijuana is present, a deep purple colored complex will result. The purple color will form if any phenolic compound with an alkyl side chain is present, e.g. resorcinol.

6. Add a small amount of chloroform to the test tube.

***If the purple complex enters the chloroform (that is it is soluble in CHCl3), the entire test is POSITIVE for the presence of Marijuana. This is a crucial part of the test sequence. If the purple color does not enter the chloroform layer, the test is considered NEGATIVE.

C. Thin Layer Chromatography (TLC)

1. The pet ether remaining in the test tube with actual plant material is spotted on to two appropriately labeled silica gel thin layer chromatography plates.
2. A marijuana standard and a blank of pet ether is also spotted on the plates.
3. Then the plates are placed into ONE of the following solvent systems:
   a. Hexane-diethlyether 4:1
   b. Benzene
   c. Chloroform
   d. Petroleum ether-methanol. 95:5 (use if suspect PCP)

   (Two systems must be used if no microscopic tests was done.)
4. Plates are visualized by spraying with a Fast Blue BB salt solution.

Separated cannabinoid components of extract will form a characteristic color:

e.g. Hexane/diethly ehter 4:1

Cannabidiol (CBD)------Orange

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Tetrahydrocannabidinol (THC)-----Scarlet
Cannabinol (CBN)-----Purple
5. Visualized spots are compared to the marijuana standard. If scarlet spot for THC is present, the test is considered POSITIVE.

A. Germination of Seeds (Done to prove manufacturing)
1. Place seeds in moist container.
2. Seeds should germinate in two the three days.
3. Seeds are considered fertile if more than 10% sprout.
4. Estimate what percentage sprouted.

II. Requirements for Specific Samples:

A. Plant material.
1. Microscopic.
2. Modified Duquenois Levine
3. TLC

B. Seeds
1. Microscopic
2. Modified Duquenois Levine
3. TLC
4. Germination

C. Hash
1. Microscopic (optional) Dissolve in $CHCl_3$ and look for crushed plant hairs.
2. Modified Duquenois Levine
3. Two different TLC systems.

D. Pipes
1. Microscopic on ash (optional)
2. Modified Duquenois Levine\
3. Two different TLC systems---only one TLC system is required if microscopic is positive.

III. CONFIRMATION OF POSITIVE:

The sample may be designated as "marijuana, non-narcotic, schedule 1" if microscopic, Duquenois and TLC are all positive.

The sample may be designated as "the resins of marijuana, non-narcotic, schedule 1" if microscopic was not done, but the Duquenois and two TLC systems were positive.

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MESCALINE ANALYSIS:

1. Dissolve in .1N H_2SO_4.
2. Wash with $CHCl_3$.
3. Make basic (pH 8) with Na_2CO_3.
4. Extract into CHCL3
5. Filter and evaporate.
6. Run IR and compare to known standard.
7. GC/MS recommendation: Column: HP 20m Ultra 1, Temperature: 100-280 degrees 20 degrees per minute for 30 minute total run time. Inlet temp: 175 degrees C. Compare retention time and ion scans to known standard.

Color test:
Marquis: orange
Mecke's: orange turning brown
Libermann's: black
Con. HNO_3: fast dark red

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Mushroom Extraction:

1. Allow mushrooms to dry if needed..
2. Grind @ 0.5 -3.0 grams of mushrooms to a fine powder.
3. Place powder in a 12.5 cm filter paper folder to fit a glass funnel.
4. Rinse mushroom powder with approx. 50ml of chloroform.
5. Discard the CHCl3
6. Allow mushroom powder to dry completely.
7. Rinse mushroom powder with approx. 50ml of 0.1N sulfuric acid COLLECT rinsings in a centrifuge tube.
8. Transfer half the extract in to another centrifuge tube (now have 25ml in two 50ml tubes)
9. Add equal volumes of CHCl3 to each of the tubes and wash (mix with lid by hand)
10. Centrifuge for about 2 minutes.
11. Remove the aqueous layers an place into two new centrifuge tubes.
12. Make the aqueous solutions basic with 1N NaOH *****Check the pH.
13. Add to the basic aqueous solutions equal volumes of methylene chlorine (Ch2Cl2).
14. Mix by hand and centrifuge for 2 minutes.
15. Aspirate the aqueous. (No need to keep, bottom layer is now of importance).
16. Transfer the extract (bottom layer) in to a 50ml beaker and boil to about 1-2ml. Do not go to dryness.
17. Transfer extract in to autoinjection vial for GC/MS. GC/MS recommendations: HP ultra 1 Column, 100-280°C, 20°/ minute, total run time 30 minutes.

For TLC plate :

18. Allow the 50 ml beaker to dry completely.
19. Reconstitute the beaker with a few drops of methanol.
20. Spot the sample directly from the beaker on to a TLC plate.
21. Run with a blank and the standard in a T1 solvent.
    T1 Solvent: 7.5 drops NH4OH and 25ml of MeOH.
    Visualize with: 1. Fast Blue BB===Red Spot
    2. p-DMAB=====Blue Spot

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Confirmation of Positive for Psilocin:

1. GCMS spectra match to psilocin standard run on the same day, along with a blank of the same solvent.
2. TLC plate with match to Rf location to known standard.

Methamphetamine/Amphetamine Acetylation Method.

1. Dissolve small amount of sample in H2O.
2. Make very basic with NaOH.
3. Extract with Petroleum Ether or Hexane.
4. Split sample in half and place in autoinjection vials.
5. Acetylate one half of the sample with 1-2 drops of acetic anhydride.
6. Run on the GC/MS; compare retention time and ion scans to known standards. GC/MS recommendations: HP Ultra 1 20m column, Temperature 100-280°C, 20°C/minute, total run time 15 minutes.

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METHAMPHETAMINE/AMPHETAMINE ANALYSIS:

1. Add .5N NaOH (2.5ml of 2N to 10ml of DI water) to a small amount of sample powder.
2. Extract with hexane (1 to 3 times ....depending on the purity of the sample)
3. Wash with H2O (1 to 3 times as necessary).
4. Dry hexane thru NaSO4.
5. Bubble with HCl fumes until precipitate forms (may use light heat for recrystallizing).
6. Filter crystals and allow to dry.
7. Run IR and compare to known standard. If poor IR is obtained, run on GC/MS. GC/MS recommendations: HP Ultra 1 Column 20m, 100-280°C, 20°C/minute, inlet temp. 175°C.

TOXI-LAB PROCEDURE:

1. Place a blank toxilab disc into a metal concentration cup.
2. Add a few drops of sample extract to the paper disc in the metal cup.
3. Allow the disc to dry with a gentile current of warm air.
5. Place the disc in to the Toxigram at the appropriate opening.
6. Allow the "loaded" Toxi-gram to warm on a hot plate for a few seconds face up.
7. Place 3ml of amine solution and 20ul of ammonium hydroxide in to a chromatography chamber and swirl vigorously for 10 seconds.
8. Remove the Tox-gram from the warmer and lower disc end first into the chromatography chamber. Do not allow the sides of the Toxi-gram to tough the walls of the chamber.
9. Remove the Toxi-gram when the dye spots on the Toxi-gram reach 9.5cm (after 12-17 minutes) and place the Toxi-gram face down on the warmer for 30-60 seconds or until the fumes have evaporated.
10. Place the Toxi-gram into a jar (A-1) that contains formaldehyde under a plastic screen which allows only the fumes to reach the Toxi-gram. Leave in the jar for 2-15 minutes.
11. Allow the Toxi-gram to warm for a few seconds on the warmer.
12. Dip the Toxi-gram slowly into concentrated sulfuric acid (A-2). Remove and note the colors.
13. Dip the Toxi-gram slowly into H20. Remove and note the colors.
14. Place the Toxi-gram under UV light, note the colors.
15. Dip the Toxi-gram unto modified dragendorff's solution, note the colors.
16. Place the Taxi-gram between two sheets of glass, make a photo copy and keep with the report.

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CONFIRMATION OF POSITIVE:

Must have IR match to a known standard .

OR

Must have GC/MS spectra match and similar retention times to a known standard and Toxilab confirmation.

Color test positives: Amphetamine............Marquis=Orange
Methamphetamine.....Marquis=Orange
.....Secondary Amine=Inky Blue

General Unknown Sample by GC/MS

1. Dilute powder, solid or residue sample in MeOH or appropriate solvent.
2. Run on GC/MS and compare to computerized library match.
3. Run standard to confirm library match, look for similar retention times and ion scans.

General Unknown Sample by IR

1. Grind powder or solid sample with KBr.
2. Form pellet and run IR scan.
3. Compare scan with library match.

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Training Guidelines

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GENERAL DRUG TRAINING GUIDELINES

I. COCAINE

A. Spot tests.
B. Direct IR.
C. Cobalt thiocyanate derivative & IR confirmation.
D. GCMS preparation & confirmation.
E. Microcrystalline test.
F. Schedule.
G. References.

II. PILLS

A. Reference search:
   1. Contents and amount.
   2. Company.
   3. Schedule.
B. Screen, confirm or no testing.
C. How to extract (reference source - Clarke).
D. Confirmation by GCMS.

III. HEROIN

A. Spot tests.
B. GCMS preparation & confirmation.
C. IR preparation & confirmation.
D. Schedule.
E. References.

IV. METHAMPHETAMINE AND AMPHETAMINE

A. Spot tests.
B. GCMS preparation & confirmation.
C. IR preparation for D meth.
D. IR preparation for DL meth by PIT derivative.
E. Microcrystalline test.
F. Schedule.
G. References.

V. GENERAL UNKNOWNS

A. Spot tests.
B. GCMS preparation.
C. IR preparation.
D. References - Clarke and Mercke.

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VI. LSD

A. Screening test/TLC.
B. GCMS preparation.
C. GCMS conversion.
D. Schedule.
E. References.

VII. MUSHROOMS

A. Spot test.
B. TLC.
C. GCMS preparation.

VIII EXTRA TRAINING AREAS

A. FTIR tutorial.
B. GCMS tutorial.
C. Microcrystalline exercise.
D. Spot test exercise.
E. TLC exercise:
   1. T1 with caffeine/ephedrine/phenylpropanolamine and cocaine/heroin/meth mixed standards - UV, fluorescamine and iodoplatinate.
   2. T1 with LSD and mushrooms - UV & PDMAB.
   3. Acetone with LSD - UV & PDMAB.
F. Drug schedule DEA exercise.
G. Reading - Clarke and all photocopied material.

IX. PROFICIENCY TEST
At least 10 unknown samples - to include cocaine, heroin, methamphetamine, and non-controlled substances.

X. MOCK COURT

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ANALYSIS - GENERAL OUTLINE

The following is a generic protocol which may be used in the analysis of a suspected drug sample. The outline may be followed by a specific protocol for the most frequently encountered drugs. It should be noted that the nature of the sample determines the analytical route the chemist pursues and that many samples do not lend themselves to an exact order of protocol. **The scientist ultimately decides the specific analytical route for each sample.**

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- **Introduction**
  It is the chemist's goal to utilize any independent analytical technique or techniques to conclusively identify an unknown substance. One of the methods used needs to provide structural data (i.e. MS or IR). A conclusive identification can be obtained when the analyte is distinguishable from all significant isomers and closely related substances, and when sources of contamination can be eliminated.

- **Physical Characterization**
  The evidence is removed from packaging, is physically described and the mass or amount determined.

- **Representative Sampling**

  In multiple exhibit cases, a representative number of exhibits may be analyzed. The method of deciding how many exhibits will be analyzed is to take the square root of the total number of exhibits.

- **Screening Tests**
  Screening tests are non-specific tests that provide preliminary data on the nature of a sample. Screening tests may consist of:
  - Chemical color tests.
  - Thin-layer chromatography
  - Gas chromatography
  - Logo identification of pharmaceuticals
  - Microscopy
  - Microcrystalline
  - UV screening

ANALYSIS - GENERAL OUTLINE (cont.)

○ SPECIFIC DRUG REFERENCE INFORMATION

A basic procedure for the analysis of a specific drug can be found in Clark's Isolation and Identification of Drugs. Since the instrumentation available in the crime laboratories is sometimes different from that in Clark's book, and sample preparation and conditions are different, it should be expected that data generated by the scientist may differ from those in the reference. For the purpose of identification of a chemical, it is expected that the scientist will compare sample (evidence) data with data gathered by use of known standards under extraction, preparation, and instrumental conditions identical to those used for the sample (evidence).

○ Sample Preparation

Many drug samples are mixtures or contain excipient material requiring the compound of interest to be separated from a matrix before subjecting the sample to further instrumental analysis. Some methods are:

☞ Solvent extraction: the analyte is dissolved in an appropriate solvent and filtered away from insoluble excipient material. The solvent is then evaporated and the analyte recrystallized.

☞ Solvent washes: the excipient material is dissolved and washed away from the analyte by using a solvent in which the analyte is insoluble.

☞ Solvent/Solvent extractions:
Two immiscible solvents can be used to extract an analyte. An aqueous phase (acidic or basic)and immiscible organic solvent phase are typically used. Many drugs are nitrogen bearing compounds that readily form salts and neutral species enabling them to be separated by acid/base and organic solvents.

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☞ Particle Picking: some mixtures and crystalline samples can be physically separated and isolated. stereomicroscope can assist in the identification of individual components of a sample and forceps or a small probe can be used to isolate them for analysis.

○ **Instrumental Analysis**

A conclusive identification of any controlled substance (except marijuana) will include sample analysis utilizing at least one of the following techniques.

☞ Infrared Spectroscopy.
☞ Mass Spectrometry.

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CLANDESTINE LAB
CASE WORK

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CLANDESTINE LAB CASE WORK

Introduction

The objective in analyzing clandestine laboratory evidence is to determine if either a purported clandestine laboratory site has the capacity to manufacture an illicit substance, or has in fact done so.

Safety Considerations

General laboratory safety guidelines are thoroughly discussed in the BFS Chemical Hygiene and Safety Plan.
Due to the hazardous nature of many of the chemicals used in clandestine laboratories, evidence received should be opened in a ventilation hood. Sample vials or containers within the outer packaging should be considered contaminated and handled with gloves. Leaking samples need to be repackaged and defective or damaged containers replaced.

Generally, care should be exercised in analyzing samples from clandestine labs for a number of reasons: the samples often come from containers which are purposely mislabeled; the samples are frequently very toxic, corrosive or flammable and sometimes highly reactive. As much information about the lab should be obtained as possible, in order to be aware of possible hazards to which the analyst may be exposed. This information can come from investigators, the crime scene report, and the individual who collected the samples. It is wise to obtain a sample inventory list, which should contain information from the observations of the chemist as the samples were taken, such as pH, color, volatility, and any identifying labels on the containers. Any documentation relating to synthesis found at the scene will also be useful in determining what the clandestine chemist was attempting to do.

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CLANDESTINE LABORATORY CASEWORK (CONT.)

○ Analysis

Clandestine lab case samples usually contain a variety of liquids and solids, pure reagents and reaction mixtures, extracts and waste chemicals. These samples may be organic or inorganic. Solutions may be aqueous, acidic or basic, or organic. Not all samples may need to be analyzed if finished products and precursors are found to be present. More likely reaction mixtures or waste with traces of finished product and other substances will be encountered.

Samples will normally be collected at the scene in duplicate to ensure that sufficient samples is available for reanalysis if required; therefore, only one sample vial per set needs to be examined. Where only one sample was taken, half or more of the sample must be preserved for reanalysis.

○ Interpretation of Results

In order to interpret the results of the analysis in this kind of case one must be familiar with at least the more common synthesis routes and procedures for the manufacture of street drugs and explosives, as these are presently the most frequently encountered clandestine substances. The identity of precursors, reagents, finished products and by-products must be considered as a whole to determine the intent of the suspect.

Comparison of the identified substances to known reaction formulas should be made. Formulas found at the scene should be examined to determine, first of all, the intent of the clandestine chemist, and secondly, if the formula is viable. This last step may require duplicating the procedure in the crime lab if it has not been documented before. In writing an opinion report, the forensic chemist must describe the evidence analyzed, the analysis methods used, and the results of the analysis. Then an opinion may be expressed regarding the suspect's capacity, intent and/or success in manufacturing an illicit substance.

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INTERPRETATION OF RESULTS (CONT.)

Not all clandestine lab evidence will contain samples which are conclusive regarding intent to manufacture. Sometimes nothing more than mineral acids, strong bases and a few organic solvents are discovered. Often many of the reagents found are common household chemicals such as drain cleaner, painting solvents or battery acid. Even specialized chemical glassware if found at the scene may not be enough in the eyes of a jury to convict an individual. The forensic chemist must be familiar with household chemicals and how they can be used by the clandestine chemist, as well as what their legitimate uses are and be able to fairly and clearly include such information in his or her report.

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Procedural Outline

An outline approach to these samples can be divided into two categories, liquids and solids. Such a scheme works also for general chemistry cases where one is asked to identify an unknown sample.

Determine if liquid sample is organic or inorganic.

If aqueous:

- Determine pH.
- Identify any anions and cations present. This may be done by wet chemistry methods or instrumental methods.
- Identify organic if present. This usually requires a solvent extraction to isolate the compound.

If organic:

- Determine if a pure solvent, and identify by IR or GC/MS.

☞ If not a pure solvent, analyze the components. Such liquid samples are likely to be reaction mixtures or extracts and may contain reaction by-products which are characteristic of a specific reaction. Analysis is usually by GC/MS.

Clandestine Lab Sample Procedural Outline (cont.)

Solid samples

Screening:

- ☞ IR - initial screening of solid samples from clandestine labs is best done by infrared spectroscopy. This precludes any adverse reactions of the sample with acidic color test and will distinguish organic from inorganic materials.
- ☞ Solubility - If the sample is soluble in methanol it may be a good candidate for GC/MS.

Identification:

- ☞ Organic solids - Best analyzed by IR, GC/MS, or a combination of the three techniques.
- ☞ Inorganic - Cannot always be identified completely, but may be at least characterized by IR, especially when observing absorbencies below 2000 cm. Wet chemistry methods of qualitative analysis can be used if sufficient sample is present.

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REVISIONS

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CHEMICAL SCREENING TESTS

Introduction

Chemical color tests are non-specific screening tests that provide preliminary data as to the nature of the substance to be identified. Certain compounds or classes of compounds produce distinct colors when brought into contact with various chemical reagents. These simple reactions can indicate the presence of a particular functional group or molecular moiety.

There is always a certain amount of subjectivity that must be taken into account when a color is reported. It is not uncommon for two analysts to describe the same color differently. Aside from the differences in reporting colors that can be attributed to the analyst, colors can also be influenced by the concentration of the sample in the reagent, by the presence of contaminants, or by the age of the reagent. Also, the length of time during which the colors are observed may influence the color reported because color transitions and instabilities are not unusual. Allowances should, therefore, be made for these differences, especially with evidence items where neither the concentration of the chemical nor the presence or composition of any contaminant is known.

Procedure

The spot tests are conducted by transferring a small amount of a substance with a clean spatula to a well on a spot plate. A drop of the chosen color reagent is then placed into the well. Any color which is observed is noted. The order of adding reagent and sample may be reversed by first placing the reagent into the spot plate well and adding the sample to the reagent.

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MASS SPECTROMETRY

Introduction

Mass spectrometry (MS) is an instrumental technique for the identification of organic compounds. Upon introducing a sample into the MS, the sample is fragmented and ionized into charged ions by bombardment with accelerated electrons. The ions are then filtered according to their mass to charge (m/z) ratio by a scanning quadrupole magnetic field. A detector and data system (computer) record the mass and quantity of ions as the spectrometer is scanning, resulting in the generation of a mass spectrum. The sample spectrum may then be compared to reference library spectra.

Procedure

The sample is introduced into the mass spectrometer through a GC equipped with a capillary column. The sample is dissolved in an appropriate solvent, injected onto the GC which separates the sample into its individual components which then enter the ion source of the MS.

Calibration and Maintenance

The mass spectrometer is calibrated and tuned by either of two means, manually or by an instrumental (computer) program which adjusts the MS operation parameters to achieve certain predefined performance criteria. The instrument shall be tuned daily before any analysis are performed. The tuning and calibration typically utilize perfluorotributyl-amine (PFTBA), a stable compound which produces ion fragments throughout the mass range for the spectrometer. These ions are used to calibrate and normalize the instrument's operation. For some work, the instrument may be tuned and calibrated using a primary standard, in order that the instrument be optimized for a particular compound. A complete discussion on tuning is found in the manufacture's operations manuals. A complete maintenance schedule can be found in the instrument's maintenance and calibration record book as well as in this manual under the instrumentation section of Drug Analysis.

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○ **Infrared Spectrometer**

A reference standard should be ran at least once a month to verify instrumentation performance, dated hard copies are maintained in log book.

Sample support media (KBr) are periodically checked to ensure they are not contaminated. The results are maintained in the log book.

Spectrum is sufficiently resolved for library comparison.

Hard copy sample data are maintained in case file.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

○ **Thin Layer Chromatography**

Solvent blank and reference standards are run with sample on each TLC plate.

Each plate must contain a standard along with any samples.

The developed TLC plates are photocopied and maintained in case file.

○ **Reporting**

The report that is issued represents a summary of the analytical findings and identifies the controlled substance or substances found and lists the item's weight. Controlled substances are reported as named in the Uniform Controlled Substances Act.

"No controlled substances". When a controlled substance is not identified the wording of the report conclusion should accurately reflect the confidence of the analysis.

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Instrumentation Quality Control Procedures

○ GC and GC/MS

GC performance (chromatography, and retention time stability) is checked by running drug standards with a hard copy of the results included in each case file. Relative retention times of samples and standards must match and mass spectrum of samples and standards must match.

A solvent blank is periodically run between case samples to insure contamination is not occurring. The number and frequency of running blanks is left to the discretion of the analyst.

The syringe is thoroughly rinsed after each injection.

All sample vials are clearly marked with item number and case number.

Unattended utilization of the autosampler on the GC/MSD require the following procedures:

- ☞ A sequence table is generated listing all samples, blanks and their position in the carrousel.
- ☞ Data file headers contain the carrousel's vial position number.
- ☞ The sequence table is checked against the vials in the carrousel prior to starting the autosample run.

Hard copy sample data are maintained in case file.

GC/MS is tuned daily when a run is to be performed and dated tune printouts are maintained in log book.

Instrumentation receives routine periodic maintenance as per manufacturer's recommendations.

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