Chain of Custody Procedure

Submitting police departments transfer evidence to our evidence officer. The evidence officer labels each item with a bar-coded lab number and enters each into the drug lab database. After all the evidence is entered for that submittal, the evidence officer generates two receipts that records the date and time. The evidence officer signs and dates both receipts. The lab keeps one copy for their records and the other copy is given to the submitting officer for their records. The evidence is placed in a bag labeled with the submitting department name on it and stored in the locked drug safe until assigned for analysis.

The evidence officer assigns samples to be analyzed to a chemist. At this time, the evidence officer scans each item to show it was assigned to the lab and to a particular chemist. A receipt with the date is generated to show the transfer to lab and the chemist and evidence officer initials the receipt. The evidence is taken to the lab for analysis and stored in a locked safe during the analysis. When analysis is complete, the evidence is returned to the evidence officer. The evidence is scanned back in to show it was returned to the drug safe. Another receipt with the date is generated that is initialed again by the chemist and evidence officer. The evidence is stored in the drug safe until the submitting department returns to pick it up.

When a department returns to pick up the evidence, the evidence is scanned back to the police officer by the evidence officer. The evidence officer generates two ‘Picked Up’ receipts with the date. The evidence officer and the officer picking up the evidence sign both receipts and the lab keeps one for their records and the other receipt is given to the officer picking up the evidence. Also at this time, all completed Drug Certificates of Analysis pertaining to the returned evidence are given to the officer.

Extended Release Amphetamine Capsules

1. Crush 5 -10 mg Amphetamine from an Adderall capsule in mortar with pestle.
2. Dissolve in 20 -25 mls. .1N HCl.
3. Filter
4. Add NH4OH until basic (1-2 drops should suffice).
5. In sepratory funnel extract with 50 mls. petroleum ether.
6. Dry the Petroleum ether extract with anhydrous Na2SO4. Add the Na2SO4 directly to the beaker (50-100mg), then filter.
7. Using a Pasteur pipette bubble HCl gas into Petroleum ether extract 2 -3 times.
8. Evaporate to dryness.
9. Add 1 drop NH4OH, dissolve in petroleum ether and add to glass autosampler vial.
10. Inject 3 – 5 ul into GC or CG/MS.

Section 1: Identification of Marijuana

I. Introduction:

Marijuana samples will be tested and analyzed by microscopic analysis, along with The Modified Duquenois Test, or The Rapid Modified Duquenois Test, as well as GC and GC/MS for samples weighing over one ounce.

II. Reagents:

A.) Modified Duquenois Test:

1. Petroleum Ether
2. Duquenois Reagent: 10 mg Vanillan, 7 mL Acetaldehyde, in 500 mL of Ethanol
3. Concentrated Hydrochloric Acid
4. Chloroform

B.) Rapid Modified Duquenois Test:

1. Duquenois Reagent: 10 mg Vanillan, 7 mL Acetaldehyde, in 500 mL of Ethanol.
2. Concentrated Hydrochloric Acid
3. Chloroform

C.) Chromatography by GC and GC/MS:

1. Petroleum Ether
2. Methanol: used for solvent rinse on the instrument

III. Equipment:

A.) Analytical Balance
B.) Microscope and slides
C.) Test Tubes
D.) Hot Plate (for when samples are moist)
E.) Porcelain Dish
F.) 2 mL Autosampler Vials with Teflon caps
G.) 10 mL Volumetric flask
H.) GC/FID: HP 6890 or 7890A
I.) GC/MS: HP 6890/5973 or HP 7890A/5975C Series.

IV. Procedure:

A.) Microscopic Test:

1. Open sample bag and remove portion of sample and place on slide.
2. Identify characteristic morphological (both macroscopic and microscopic) features of Cannabis sativa by concentrating on leaves, small twigs, seed hulls, cystolith hairs, glandular hairs, and flowering tops.
3. Both cystolith hairs and glandular hairs should be observed to be considered positive.

B.) Modified Duquenois Test:

1. Extract 30-100 mg of sample with 15-20 mL of Petroleum Ether
2. Filter and evaporate the filtrate in a white porcelain dish.
3. Add 2mL of Duquenois reagent and stir to bring residue into solution.
4. Add 2 mL of concentrated Hydrochloric Acid, stir and let stand 10 minutes. A color will develop.
5. Transfer colored solution to labeled test tube and shake with 1-2 mL of Chloroform.
6. If marijuana is present in the sample, the violet color will be transferred to the Chloroform layer.

C.) Rapid Modified Duquenois Test:

1. Place 25-60 mg of dry crushed marijuana in a test tube and shake with 2 mL of Duquenois reagent for 1 minute.
2. Add an equal amount of concentrated Hydrochloric Acid and observe the color changes to a final violet shade.
3. Shake the mixture with 1-2 mL Chloroform.
4. If marijuana is present in the sample the violet shade will be transferred to the Chloroform layer.

D.) Residues and Smoking Apparatus's (SMAPPS):

1. Solvent rinse the apparatus containing sample or residue with Petroleum Ether.
2. Evaporate off the solvent to about 1-2 mL.
3. Transfer this solvent to a 2 mL autosampler vial to be run on GC/MS.
4. Evaporate off any leftover solvent until dry.
5. Proceed with the Modified Duquenois Test (IV.B or IV.C)

E.) Chromatography by GC and GC/MS:

1. For marijuana samples that are over 1 ounce, residues and smoking apparatus's, samples will be run on GC followed by GC/MS for confirmation.
2. Add between 30-100 mg of sample to an autosampler vial, enough so the bottom of the vial is covered.
3. Add 1-2 mL of Petroleum Ether to the autosampler vial, cap and shake a few times.
4. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
5. GC/FID conditions are as follows:

Method: EXP.M
Oven:

Initial Temp: 245°C
Initial Time: 0.00 min.
Rate: 10°/min.
Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.

Inlet:

Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium
Column:

Capillary: HP-1 30m x 320um

Initial Flow: 3.3 mL/min.

Detector:
Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium

6. Obtain chromatographs. If cocaine is present, the instrument will detect a peak with a retention time around 3.40 minutes and will generate a report with accompanying chromatograph.
7. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
8. Place same sequence on the GC/MS autosampler and run.
9. GC/MS conditions:

Method: EXP
Oven:
Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibrium Time: 0.50 min.
Rate: 10.0°/sec
Final Temp: 280°C
Run Time: 10.00 min.
Inlet:
Injector Temp: 250°C
Mode: Split
Pressure: 31.65 psi
Gas Type: Helium
Column:
Initial Flow: 1.0 mL/min.
Column: Capillary (30m long, 320 um diameter) HP1MS

10. If THC is present in the sample, the GC/MS will detect the peak for delta-9-THC, and will generate a report with chromatograph. delta-9-THC data is stored in the GC/MS

library and is used to confirm hits detected on the GC/MS. The library can also be accessed directly without needing to run a sample first. (See graph, last page).

E.) THC Quantitation Procedure:

1. Make Stock Solution: $C_{30}H_{64}$, 2 mg/mL in Petroleum Ether
2. Make Working Solution by transferring 5 mL of stock solution to a 10 mL volumetric flask, add 1 mL THC standard (10 mg/mL in Ethanol), and bring to volume with Petroleum Ether.
3. Prepare sample by measuring 400 mg of sample and soaking in Petroleum Ether overnight. Next, filter and evaporate to about 3 mL (do not let go dry). In a 10 mL volumetric flask, add 5 mL of stock solution, 3 mL of sample, then rinse container containing the sample with Petroleum Ether to bring volume volumetric flask to volume.
4. Transfer solutions mentioned above to appropriately labeled autosampler vials and run on GC. The sequence run is as follows: THC STD., THC STD (Calib.), THC STD., BLK, SAMPLE (S), THC STD.
5. Calculations for % THC:

$$\frac{\text{PK HT THC(sample)}}{\text{PK HT}(C_{30}H_{64})} \times \frac{\text{PK HT}(C_{30}H_{64}\text{ STD})}{\text{PK HT THC(STD)}} \times \frac{\text{mg THC(STD)}}{\text{mL(STD)}} \times \frac{\text{tot.vol sample(mL)}}{\text{wt.sample (mg)}} \times 100\%$$

V. Results:

A.) For the Duquenois Tests, record all results, including date, sample number, and results on the Drug Lab Results sheet that are with samples.

B.) The results from the Drug Lab Results sheets will then be reported to the submitting agency on formal Certificate of Analysis.

C.) When performing analysis on GC and GC/MS, print out results with chromatographs, and report as stated above. File chromatographs so they may be accessed if necessary.

Section 2: Identification of Cocaine and Crack

I. Introduction:

Cocaine and Crack samples will be analyzed by a microcrystalline test and run on GC/FID followed by confirmation on GC/MS. The microcrystalline test utilizes TLTA (di-p-toluoyl-l-tartaric acid) as a reagent. For cocaine samples, TLTA alone will produce crystallization. However, for crack samples, TLTA and Acetic Acid is used to produce crystallization. The purpose of the microcrystalline test is to determine the presence of the l-cocaine diastereoisomer.

II. Reagents:

A.) TLTA (di-p-toluoyl-l-tartaric acid): 10 mg TLTA, 1mL Ethanol, 1mL glycerine in 8mL distilled water.
B.) Acetic Acid (HOAC)
C.) 9:1 Methylene Chloride/Isopropanol, Ethanol, or Methanol.
D.) Methanol (solvent rinse for GC)
E.) 0.1 N HCL: add 8.3mL concentrated HCL to sufficient water to make 1 L. (quantitation).
F.) Cocaine HCL in $C_{24}H_{50}$ (quantitation standard).
G.) 10% K_2HPO_4: dissolve 10g K_2HPO_4 in sufficient water to make 100mL. (quantitation).
H.) $C_{24}H_{50}$ in Methylene Chloride (quantitation internal standard).
I.) Anhydrous Sodium Sulfate (Na_2SO_4).

III. Equipment:

A.) Microscope and slides
B.) Analytical balance
C.) Weigh paper
D.) Pipettes
E.) 50 mL volumetric flask
F.) 25 mL volumetric flask
G.) Stoppered test tubes (2)
H.) 2 mL autosampler vials with Teflon caps
I.) GC/FID: HP 6890 or 7890A
J.) GC/MS: HP 7890A/5975C or HP 6890/5973 series.

IV. Procedure:

A.) Chromatography by GC/FID and GC/MS.

1. Obtain gross weight of sample evidence bag.
2. Remove one sample packet from the evidence bag and weigh.
3. Record individual packet gross weight in logbook.
4. Remove substance from packet onto a weigh paper.
5. Reweigh the empty packet and record the weight.
6. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
7. Return remaining substance back to packet and reseal
8. Return packet(s) to original evidence back, reseal, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the substance's net weight.
10. For up to 100 packets, analyze 10% of the total. Report the statistical average of the individual net weights.
11. For more than 100 packets, analyze the square root of the total. Report the statistical average of all the individual net weights.
12. Add 1-2 mL of Ethanol, Methanol or 9:1 Methylene Chloride/Isopropanol to the vial and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
    GC/FID conditions are as follows:

    Method: EXP.M
    Oven:
    Initial Temp: 245°C
    Initial Time: 0.00 min.
    Rate: 10°/min.

Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.
Inlet:
Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium
Column:
Capillary: HP-1 30m x 320um
Initial Flow: 3.3 mL/min.
Detector:
Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium

14. Obtain chromatographs. If cocaine is present, the instrument will detect a peak with a retention time around 3.40 minutes and will generate a report with accompanying chromatograph.
15. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
16. Place same sequence on the GC/MS autosampler and run.
17. GC/MS conditions are as follows:
    Method: EXP.M
    Oven:
    Initial Temp: 230°C
    Initial Time: 0.00 min.
    Max. Temp: 325°C
    Equilibration Time: 0.50 min.
    Rate: 10°/min.
    Final Temp: 280°C
    Run Time: 10 min.
    Inlet:
    Mode: split (50:1)
    Initial Temp: 250°C
    Pressure: 31.65 psi
    Gas Type: Helium
    Column:
    Capillary: HP-1MS 25m x 200um x 0.33um
    Max. Temp: 300°C
    Initial Flow: 1.0 mL/min.

18. If cocaine is present in sample, the instrument will detect a total ion peak at approximate retention time of 3.50 minutes and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak, its ion abundance and the ratio of ions 152:150, confirming the presence or absence of cocaine and the l-cocaine diastereoisomer. (see graph, last page)

B.) Microcrystalline Test:

1. On a microscope slide add a small amount of sample.
2. Add one drop of TLTA reagent to sample.
3. Crystallization should occur within a few minutes if the l-cocaine diastereoisomer is present.
4. If the sample does not crystallize within a few minutes, add one drop of HOAC to the sample on the slide. Observe under microscope at same magnification. Crystallization should occur within a few minutes, indicating that the sample is the free-base form of cocaine (crack).

C.) Quantitation Procedure:

1. Extract sample by weighing out 100 mg of sample in a 50 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.
3. Prepare cocaine standard by weighing out 25 mg of Cocaine HCL in a 25 mL volumetric flask and bringing to volume with $C_{24}H_{50}$ in Methylene chloride.
4. In first stoppered test tube, labeled standard, add 2 mL of standard solution (prepared in step#3), 2 mL of 0.1 N HCL, and 1 mL of K_2HPO_4.
5. In second stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K_2HPO_4. Two layers will form in each tube.

6. In labeled autosampler vials, add enough sodium sulfate (Na_2SO_4) to cover the bottom of the vial.
7. From each of the test tubes, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Cocaine Standard (inj.#1), Cocaine Standard (inj.#2, Calibration), Cocaine Standard (inj.#3), Blank, Sample(s), Cocaine Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

V. Results:

A.) For the microcrystalline test, record in logbook as TLTA positive, TLTA positive with HOAC, TLTA negative, or TLTA negative with HOAC.
B.) Record results of the GC/MS in logbook. Then transfer the results to Drug Lab Result sheet that came with the actual samples. Be sure to include date of analysis, results, the number of packets analyzed, and signature.
C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

VI. Discussion:

When analyzing for cocaine, one must take into consideration the four diastereomers of cocaine. The four diastereomers are cocaine, pseudococaine, allococaine, and pseudoallococaine. Diastereomers are completely different chemical entities. All their measurable physical characteristics are different. These differences are measured by a number of different analytical methods. The one we use here is a microcrystalline test along with a GC/MS ion ratio. The ion ratios are different for each diastereomer. For cocaine, the ratio is between 1 and 2, for pseudococaine its 5 to 7, for allococaine its 7to10, and for pseudoallococaine its 3 to 5.
The cocaine diastereomer is what is important to look for. It has two mirror image forms called l-cocaine and d-cocaine. The l-cocaine diastereomer is the form considered illegal. So for legal purposes, the sample must be analyzed for the presence of the l-cocaine

diastereomer. First, the microcrystalline test is used. The l-cocaine is confirmed when crystallization occurs with the addition of TLTA to the sample. If the sample is the free-base form of cocaine, acetic acid must be added along with TLTA to produce crystallization. Secondly, analysis by GC/MS will confirm the presence of l-cocaine by calculating the ion ratio of ions 152/150. If the result is less than two, the sample is positive for the presence of l-cocaine and is therefore illegal to posses.

Q.C. Procedure I: Analytical Balance Calibration

I. Introduction:

Balances in the Drug Laboratory are to be calibrated every 30 days and professionally cleaned and calibrated every year by an outside technician. There are two balances per bench (Benches 1A, 1B, 2A, 2B), the FX-2000 and the Explorer (analytical balance). For the FX-2000 a standard mass of 200g is used for calibration. To check for accuracy before and after the balances are calibrated, a series of known masses (1.0g, 2.0g, 5.0g, 10.0g, 20.0g, 50.0g) are used. These masses are from the Permas metric weights, Class S, distributed by Fisher Scientific. When not in use, the masses must be stored in a secure, cool and dry place. Handle all weights with non-scratching forceps.

II. Procedure

A.) Optimal Conditions for Weighing:

1. Make sure balance is level and on a solid surface.
2. Optimal room temperature is about 28°C/68°F with a relative humidity of about 50%.
3. Keep laboratory and immediate area around balance clean and dry.
4. Corners of a room are best due to fewer vibrations.
5. Balances should not be installed near heaters, air conditioners, or in direct sunlight.
6. Any equipment containing magnets should be kept away from balances.
7. Use a breeze break to keep out drafts.
8. Ensure a stable AC power supply.
9. Use a clean damp cloth to clean, do not use solvents.
10. Warm up balance before using or leave on standby overnight.

B.) Quality Control

1. For each of the balances, weigh each of the known masses (1.0g, 2.0g,5.0g,10.g, 20.0g,50.0g) and record results in a QC notebook.
2. The margin of error for the FX-2000 is +/- 0.02g and +/- 0.0002g for the Explorer. Therefore, if the results fall outside these margins, the balances must be calibrated and masses reweighed.

C.) Calibration

1. FX-2000 Balance

    a.) Slide rear calibration switch up to “ON” position
    b.) When “CAL O” is displayed, press RE-ZERO.
    c.) After a pause, “CAL F” will be displayed and the 200g mass can be placed on the pan.
    d.) Press RE-ZERO and after another pause, “CAL END” will be displayed.
    e.) Slide rear calibration switch down, the calibration is over.

2. Explorer Balance

    a.) Press SETUP, “CAL” should be displayed.
    b.) Press ENTER three times until “INCAL” is displayed on the screen.
    c.) Allow balance to calibrate itself, it should only take a minute or two, during which time you should hear the masses moving.
    d.) When the calibration is complete, the balance should return automatically to the “WEIGH” mode.

III. Results:

A.) All results must be recorded in appropriate QC logbook assigned for all balances on each working lab bench.
B.) This calibration process may be repeated up to 6 times, recoding each result. If the balance(s) still do not meet requirements, the balance(s) must be serviced and/or repaired.
C.) Records must be kept of all service dates, findings, and repairs in a bound logbook for each balance.

Section 3: Identification of Heroin

I. Introduction:

Heroin samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS.

II. Reagents:

A.) 9:1 Methylene Chloride/Isopropanol, Ethanol, or Methanol.
B.) Methanol (solvent rinse for GC)
C.) 0.1 N HCL: add 8.3 mL concentrated HCL to sufficient water to make 1 L. (quantitation).
D.) Benzopinacolone and 1.0 mg/mL heroin (quantitation standard).
E.) 10% K_2HPO_4: dissolve 10g K_2HPO_4 in sufficient water to make 100 mL. (quantitation).
F.) 1.0 mg/mL Benzopinacolone in Methylene chloride (quantitation internal standard).
G.) Anhydrous Sodium Sulfate (Na_2SO_4).

III. Equipment:

A.) Analytical balance
B.) Weigh paper
C.) Pipettes
D.) 25 mL volumetric flask
E.) Stoppered test tube
F.) 2 mL autosampler vials with Teflon caps
G.) GC/FID: HP 6890 or 7890A
H.) GC/MS: HP 7890A/5975C or HP 6890/5973 series.

IV. Procedure:

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A.) Chromatography by GC/FID and GC/MS

1. Obtain gross weight of sample from evidence bag.
2. Remove one sample packet from the remaining sample packets in the evidence bag and weigh.Record individual packet gross weight in logbook.

3. Remove powder from packet onto a weigh paper.
4. Reweigh the empty packet and record the weight.
6. Add about 5 mg of sample to a labeled 2mLautosampler vial.
7. Put remaining powder back into packet and reseal.
8. Return packet(s) to original evidence bag, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the powder's net weight for the sampled packet.
10. For up to 100 packets, analyze 10% of the total. Report the average of the individual the net weights.
11. For more than 100 packets, analyze the square root of the total. Report the average of all the individual net weights.
12. Add 1-2 mL of Methanol, Ethanol or 9:1 Methylene Chloride/Isopropanol to the autosampler vial(s) containing the sample and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
14. GC/FID conditions are as follows:
    Method: EXP.M
    Oven:
        Initial Temp: 245°C
        Initial Time: 0.00 min.
        Rate: 10°/min.
        Final Temp: 290°C
        Run Time: 10 min.
        Max. Temp: 325°C
        Equilibration Time: 0.5 min.
    Inlet:
        Mode: split (35:1)
        Initial Temp: 250°C
        Pressure: 24.99 psi
        Gas Type: Helium
    Column:
        Capillary: HP-1 30m x 320um

Initial Flow: 3.3 mL/min.

Detector:

Temp: 300°C

Hydrogen Flow: 30.0 mL/min.

Air Flow: 400 mL/min.

Makeup Gas: Helium

15. Obtain chromatographs. If heroin is present, the instrument will detect a peak and will generate a report with accompanying chromatograph.
16. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.
17. Place same sequence on the GC/MS autosampler and run.
18. GC/MS conditions are as follows:

Method: EXP.M

Oven:

Initial Temp: 230°C

Initial Time: 0.00 min.

Max. Temp: 325°C

Equilibration Time: 0.50 min.

Rate: 10°/min.

Final Temp: 280°C

Run Time: 10 min.

Inlet:

Mode: split (50:1)

Initial Temp: 250°C

Pressure: 31.65 psi

Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um

Max. Temp: 300°C

Initial Flow: 1.0 mL/min.

19. If heroin is present in sample, the instrument will detect a total ion peak and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

B.) Quantitation Procedure (if required):

1. Extract sample by weighing out 100 mg of sample in a 25 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.
3. The heroin standard is prepared and should contain about 1.0 mg/mL of Benzopinacolone and 1.0 mg/mL of heroin.
4. The internal standard is 1.0 mg/mL Benzopinacolone in Methylene Chloride.
5. In a stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K_2HPO_4. Two layers will form in tube.
6. In a labeled autosampler vial, add enough sodium sulfate (Na_2SO_4) to cover the bottom of the vial.
7. From the sample test tube, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Heroin Standard (inj.#1), Heroin Standard (inj.#2, Calibration), Heroin Standard (inj.#3), Blank, Sample(s), Heroin Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

V. Results:

A.) Record results of the GC/MS in logbook. Then transfer the results to the Drug Lab Results sheet that came with the samples. Be sure to include date of analysis, results, the number of packets analyzed, and signature.

B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section 4: Identification of Hallucinogenic Mushrooms

I. Introduction:

Hallucinogenic mushrooms are first given a visual test to check for their characteristic bluish-green striations in the stems. Then the mushroom sample are solvent extracted using Ethyl Ether. This extract is then run on the GC/MS. If the mushroom sample is a hallucinogen, psilocin will be detected on the GC/MS.

II. Reagents:

A.) 3 N Acetic Acid (HOAC): 37.5 mL glacial acetic acid in 412.5 mL distilled water.
B.) Ethyl Ether
C.) Ammonium Hydroxide (NH_4OH).
D.) Methanol
E.) Anhydrous Sodium Sulfate (Na_2SO_4).

III. Equipment:

A.) 250 mL glass beakers (4).
B.) Mortar and pestle.
C.) 7.0 cm filter paper.
D.) 250 mL volumetric vacuum flask.
E.) Glass separatory funnels with stoppers and stopcocks (2).
F.) Glass stir rod.
G.) pH paper.
H.) Glass pipettes.
I.) Hot sand bath.
J.) 5 uL autosampler syringe (for manual injections)
K.) GC/MS: HP 5890/5972 series.

IV. Procedure:

A.) Extraction Using Ethyl Ether:

1. Observe stems of the mushroom sample for bluish-green striations.
2. Weigh out 1.0 g of sample in a tared 250 mL beaker. Be sure to include all parts of the mushroom (caps and stems).
3. Grind the sample into finer pieces with a mortar and pestle. Add back to the beaker.

4. Fill beaker with 3 N HOAC so solution is just above the sample and so sample is saturated. Let sit overnight in a dark place (ex. drawer) because light will break down the psilocin. For quicker extractions, the beaker may be heated at 80° for one hour.
5. Vacuum filter with 3 N HOAC, making sure to rinse the beaker very well.
6. In a hood, extract with ethyl ether by pouring the filtered sample into a glass separatory funnel, add ethyl ether (5:1 organic to aqueous), stopper and invert a several times. Make sure to vent the funnel to let excess gas inside the funnel escape.
7. Remove stopper and allow the layers to separate. The ethyl ether layer will be on the top.
8. Drain the bottom layer directly into another glass separatory funnel and discard the ethyl ether layer into appropriate waste container.
9. Add NH_4OH until solution is basic.
10. Extract again with ethyl ether (5:1 organic to aqueous). This time discard the bottom layer into waste container and drain the ethyl ether layer through a funnel filled with Anhydrous Na_2SO_4 (to remove any water) into a beaker labeled with the sample number.
11. Either let the sample evaporate overnight in a dark place, or place on a hot sand bath until dry.
12. Rinse the beaker with Methanol, swirling to make sure the sides of the beaker get hit with Methanol.

B.) Chromatography by GC/MS:

1. Manually inject, in the following order, 1.5 uL of the psilocin standard, blank, and sample(s) directly from the beaker into the GC/MS.
2. GC/MS conditions:
   Method: PSILM.M
   Oven:
   - Initial Temp: 230°C
   - Initial Time: 0.00 min.
   - Max. Temp: 325°C
   - Equilibration Time: 0.50 min.
   - Rate: 10°/min.
   - Final Temp: 280°C
   - Run Time: 10 min.

   Inlet:
   - Mode: split (50:1)

Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

3. If psilocin is present in sample, the instrument will detect a total ion peak at its retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance (see graph, last page).

V. Results:

A.) Record results of the GC/MS in logbook. Then transfer the results to the Drug Lab Results sheet that came with the samples. Be sure to include date of analysis, results, the number of tests performed per sample, and signature.

B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section 5: Identification of Amphetamines and Methamphetamines

I. Introduction:

Amphetamines, methamphetamines and related Phenethylamines are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series. The samples are extracted by a simple solvent extraction procedure. However, an extraction procedure specific for non-volatile organic poisons can also be used (See Section IX).

II. Reagents:

A.) Petroleum ether
B.) Ammonium hydroxide (NH_4OH)
C.) Methanol (GC solvent rinse)
D.) 9:1 Methylene Chloride/NH_4OH

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 7890A
E.) GC/MS: HP 6890/5973 or HP 7890A/5975C series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the

computer's description matches your sample's description (See example, last page).

7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code (such as a picture) or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 9:1 Methylene chloride/NH_4OH.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples).
5. GC/FID conditions are as follows:

    Method: EXP.M
    Oven:
    Initial Temp: 245°C
    Initial Time: 0.00 min.
    Rate: 10°/min.
    Final Temp: 290°C
    Run Time: 10 min.
    Max. Temp: 325°C
    Equilibration Time: 0.5 min.
    Inlet:
    Mode: split (35:1)
    Initial Temp: 250°C
    Pressure: 24.99 psi
    Gas Type: Helium
    Column:
    Capillary: HP-1 30m x 320um
    Initial Flow: 3.3 mL/min.
    Detector:
    Temp: 300°C
    Hydrogen Flow: 30.0 mL/min.
    Air Flow: 400 mL/min.
    Makeup Gas: Helium

6. Obtain chromatographs. If sample contain amphetamine, methamphetamine, or any other phenethylamines, the instrument will detect a total ion peak with a retention time characteristic of that compound and

will generate a report with accompanying chromatograph.

7. Depending on results, analyst may want to rerun the sample using Petroleum Ether/NH_4OH.
8. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
9. Amphetamines and Methamphetamines are early eluents so a different method will need to be run on the GC/MS that allows for a slower run time.
10. Place same sequence ran on the GC/FID on the GC/MS autosampler and run.
11. GC/MS conditions are as follows:
    Method: HYD.M
    Oven:
    Initial Temp: 130°C
    Initial Time: 2.50 min.
    Max. Temp: 325°C
    Equilibration Time: 0.50 min.
    Rate: 10°/min.
    Final Temp: 280°C
    Run Time: 30 min.
    Inlet:
    Mode: split (50:1)
    Initial Temp: 250°C
    Pressure: 31.65 psi
    Gas Type: Helium
    Column:
    Capillary: HP-1MS 30m x 0.530mm
    Max. Temp: 300°C
    Initial Flow: 1.0 mL/min.
12. If amphetamine/methamphetamine is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to Drug Lab Results sheet that came with the samples. Be sure to include date of analysis, results, the number of tests performed per sample, and signature.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section 6: Identification of Barbiturates

I. Introduction:

Barbiturates are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series. The samples are extracted by a simple solvent extraction procedure. However, an extraction procedure specific for non-volatile organic poisons can also be used (See Section IX).

II. Reagents:

A.) Petroleum ether
B.) Methanol (GC solvent rinse)
C.) 9:1 Methylene Chloride/NH_4OH

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2 mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 7890A series
E.) GC/MS: HP 6890/5973 or HP 7890A/5975C series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the computer's description matches your sample’s description.

7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 1-2 mL of 9:1 Methylene Chloride/NH_4OH to vial and cap.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples).
5. GC/FID conditions are as follows:
   Method: EXP.M
   Oven:
       Initial Temp: 245°C
       Initial Time: 0.00 min.
       Rate: 10°/min.
       Final Temp: 290°C
       Run Time: 10 min.
       Max. Temp: 325°C
       Equilibration Time: 0.5 min.
   Inlet:
       Mode: split (35:1)
       Initial Temp: 250°C
       Pressure: 24.99 psi
       Gas Type: Helium
   Column:
       Capillary: HP-1 30m x 320um
       Initial Flow: 3.3 mL/min.
   Detector:
       Temp: 300°C
       Hydrogen Flow: 30.0 mL/min.
       Air Flow: 400 mL/min.
       Makeup Gas: Helium
6. Obtain chromatographs. If sample contains any barbiturates the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.

7. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
8. Barbiturates are run with a different method on the GC/MS. This method analyzes for hydrocarbons and allows for a slower run time to detect early eluters.
9. Place the same sequence that ran on the GC/FID on the GC/MS autosampler.
10. GC/MS conditions are as follows:
    Method: HYD.M
    Oven:
        Initial Temp: 130°C
        Initial Time: 2.50 min.
        Max. Temp: 325°C
        Equilibration Time: 0.50 min.
        Rate: 10°/min.
        Final Temp: 280°C
        Run Time: 30 min.
    Inlet:
        Mode: split (50:1)
        Initial Temp: 250°C
        Pressure: 31.65 psi
        Gas Type: Helium
    Column:
        Capillary: HP-1MS 30m x 0.530mm
        Max. Temp: 300°C
        Initial Flow: 1.0 mL/min.
11. If any barbiturate is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook, as well as on the evidence cards that came with the samples. Be sure to include date of analysis, results, and initials. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the

actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section 9: Extraction of Non-Volatile Organic Poisons

I. Introduction:

This procedure allows for the extraction and separation of organic poisons into their acidic, basic, or neutral forms. This procedure uses a direct extraction approach for tablets, capsules, powders, and beer. This extraction procedure can be used to separate aspirin (strong acid, A1 fraction) from codeine (basic, D fraction) as well as extract barbiturates (weak acids, A2 fraction), hallucinogens such as PCP (weak acid, A2 fraction), amphetamines (basic, D fraction), and narcotic analgesics such as oxycodone (basic, D2 fraction).

II. Reagents:

A.) Ether
B.) Ammonium hydroxide (NH_4OH)
C.) Chloroform ($CHCl_3$)

III. Equipment:

A.) Glass funnel
B.) Filter paper
C.) Beaker, sizable to contain filtrate amount
D.) pH paper
E.) Glass stir rod

IV: Procedure:

A.) Direct Extraction (tablets, capsules, powders, beer)

1. Acidify liquid samples and collect filtrate.
2. Extract solids samples first with acidulated water and collect filtrate.
3. Extract filtrate (from step #1 or #2) with ether. There will be two fractions that develop: an Ether "A" fraction (Acid Ether Extract) that contains all ether-soluble acidic and neutral drugs (such as aspirin and barbiturates) and an Aqueous fraction that requires further extraction.
4. Make the Aqueous fraction alkaline with NH_4OH.
5. Extract aqueous fraction with chloroform.
6. Again, two fractions are formed. One fraction is the Chloroform "D" fraction (Basic Chloroform Extract)

that contains all chloroform-soluble basic drugs (such as amphetamines and codeine) and the other fraction is an aqueous fraction that contains water soluble, ether insoluble, and chloroform insoluble drugs.

NOTE: The Acid Ether Extract or “A” Fraction has 3 subgroups:

1. Strong Acid drugs
2. Weak acid or phenolic drugs
3. Neutral drugs

The Basic Chloroform Extract or Chloroform “D” Fraction also has 3 subgroups:

1. Basic drugs with chloroform soluble salts
2. Basic non-amphoteric drugs
3. Amphoteric drugs

Section VIII: Identification of Phencyclidine (PCP)

I. Introduction:

Phencyclidine or PCP is screened by a microcrystalline test and then analyzed by GC/FID and GC/MS. The samples are extracted by a simple solvent extraction procedure. However, extraction by The ANOR Procedure (Section X) or extraction for non-volatile organic poisons (Section IX) can also be used.

II. Reagents:

A.) Petroleum ether or 9:1 Methylene chloride/Isopropanol
B.) Gold Chloride ($AuCl_4$)
C.) Methanol (GC solvent rinse)

III. Equipment:

A.) Analytical balance
B.) Weigh paper
C.) Microscope and slides
D.) 2 mL autosampler vials with Teflon caps
E.) GC/FID: HP 6890 or 7890A series
F.) GC/MS: HP 6890/5973 or HP 7890A/5975C series

IV. Procedure:

A.) Microcrystalline Test:

1. On a microscope slide add a small amount of sample.
2. Add one drop of $AuCl_4$ reagent to sample.
3. Observe under microscope at 100-125X. Crystallization should occur within a few minutes if phencyclidine is present.

B.) Chromatography by GC/FID and GC/MS

1. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
2. Add 1-2 mL of Ethanol or 9:1 Methylene Chloride/Isopropanol to the vial and cap.
3. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
4. GC/FID conditions are as follows:
   Method: EXP.M
   Oven:
   Initial Temp: 245°C
   Initial Time: 0.00 min.
   Rate: 10°/min.

Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.

Inlet:
Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium

Column:
Capillary: HP-1 30m x 320um
Initial Flow: 3.3 mL/min.

Detector:
Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium

1. Obtain chromatographs. If sample contains PCP the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
2. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
5. Place same sequence on the GC/MS autosampler and run.
6. GC/MS conditions are as follows:
   Method: EXP.M
   Oven:
   Initial Temp: 230°C
   Initial Time: 0.00 min.
   Max. Temp: 325°C
   Equilibration Time: 0.50 min.
   Rate: 10°/min.
   Final Temp: 280°C
   Run Time: 10 min.
   Inlet:
   Mode: split (50:1)
   Initial Temp: 250°C
   Pressure: 31.65 psi
   Gas Type: Helium
   Column:
   Capillary: HP-1MS 25m x 200um x 0.33um
   Max. Temp: 300°C
   Initial Flow: 1.0 mL/min.
3. If PCP is present in the sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will

accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

V. Results:

A.) For the microcrystalline test, record in logbook as positive, or negative with $AuCl_4$.

B.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section VII: Identification of Opiates

I. Introduction:

Opiates and its derivatives are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series. The samples are extracted by a simple solvent extraction procedure. However, extraction by The ANOR procedure can also be used (See Section X).

II. Reagents:

A.) Petroleum ether
B.) Methanol (GC solvent rinse)
C.) 9:1 Methylene Chloride/NH_4OH

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2 mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 7890A series
E.) GC/MS: HP 6890/5973 or HP 7890A/5975C series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the computer's description matches your sample's description.
7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 1-2 mL of 9:1 Methylene Chloride/NH_4OH to vial and cap.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples, STD).
5. GC/FID conditions are as follows:
   Method: EXP.M
   Oven:
      Initial Temp: 245°C
      Initial Time: 0.00 min.
      Rate: 10°/min.
      Final Temp: 290°C
      Run Time: 10 min.
      Max. Temp: 325°C
      Equilibration Time: 0.5 min.
   Inlet:
      Mode: split (35:1)
      Initial Temp: 250°C
      Pressure: 24.99 psi
      Gas Type: Helium
   Column:
      Capillary: HP-1 30m x 320um
      Initial Flow: 3.3 mL/min.
   Detector:
      Temp: 300°C
      Hydrogen Flow: 30.0 mL/min.
      Air Flow: 400 mL/min.
      Makeup Gas: Helium
6. Obtain chromatographs. If sample contains any opiates or opiate derivatives the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
7. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
8. Place the same sequence that ran on the GC/FID on the GC/MS autosampler.
9. GC/MS conditions are as follows:
   Method: EXP.M
   Oven:
      Initial Temp: 230°C

Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.

Inlet:

Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

10. If opiates are present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook, as well as on the evidence cards that came with the samples. Be sure to include date of analysis, results, and initials. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section XI: Alcohol Quantitation

I. Introduction:

Alcohol samples are analyzed and quantitated by GC/FID.

II. Reagents:

A. N-Propanol (internal standard and blank)
B. 5.00% Ethanol (standard)
C. Methanol (solvent rinse)

III. Equipment:

A. 25 mL volumetric flask (2)
B. Glass pipettes
C. 2 mL autosampler vials with Teflon caps
D. GC/FID: HP 5890

IV. Procedure:

A. Prepare 4.75% Ethanol standard in a 25 or 50 mL volumetric flask.
B. Transfer 1-2 mL to an autosampler vial and cap.
C. Prepare N-Propanol Blank and add to an autosampler vial.
D. In a 25 mL volumetric flask, add 1 mL of N-Propanol.
E. Fill to volume with the liquid sample and invert to mix solution.
F. Transfer sample to an autosampler vial and cap. (NOTE: depending on suspect sample, dilutions may be necessary).
G. Place the vials on the autosampler with the following sequence: Standard, Blank, Sample, Standard. The standard will be run 3 times for calibration purposes, then again at the end as a reference/quantitation. Also, the sample will be run twice for result accuracy and verification.

H. The GC/FID conditions are:
   Method: ALC5%.MTH
   Oven:
      Initial Temp: 35°C
      Initial Time: 3.50 min.
      Max. Temp: 120°C
      Rate: 0.0 ^{o}/min.
      Inlet: (front injector only)
         Inlet Temp: 150°C
         Gas Type: Helium
      Detector: (front detector only)

Temp: 200°C
Makeup Gas: Helium
Column:
HP WAX 15m x 0.25mm x 0.25um

I. If alcohol id present, the instrument will detect a peak at approximately 1.1 minutes for ethanol (alcohol) and approximately 2.0 minutes for N-Propanol. A report will be generated along with accompanying chromatograph (See graph, last page). The report will contain the percentage of each analyte.

V. Results:

A. Make sure that the results for all the standard runs are consistent with each other as well as consistent with the known percentage of alcohol in the standard (approximately 5%).
B. Record the average alcohol percentage of the two sample runs in the logbook and transfer the results to appropriate evidence cards, as well as any paperwork that came with the sample. Be sure to include the date of analysis, result, and initials on the evidence cards.
C. All reports generated from the instrument should be filed so they may be accessed at a later date, if necessary.

Section XII: Identification of Lysergic Acid Diethylamide (LSD)

I. Introduction:

LSD samples are analyzed by GC/MS. When sample amount permits, screen samples by Thin Layer Chromatography (TLC).

II. Reagents:

A. Ethanol
B. Methanol
C. Ammonium Hydroxide (NH_4OH)
D. LSD Standard

III. Equipment:

A. Glass microvials.
B. 2 mL autosampler vials and Teflon caps.
C. Tweezers
D. Glass cutter
E. 10 uL autosampler syringe
F. 100 mL graduated cylinder
G. Glass evaporation beaker
H. TLC Plate ($CaCO_3$)
I. TLC Chamber
J. Model CC-80 UV Fluorescence Analysis Cabinet
K. GC/MS: HP 5890/5972 series.

IV. Procedure:

A. Screening by Thin Layer Chromatography (TLC):

1. Prepare TLC chamber by making a solution of NH_4OH and Methanol. Add 1.5 mL of NH_4OH into a 100 mL graduated cylinder and bring to volume with Methanol. Add solution to the TLC Chamber.
2. Place the sample into a glass evaporation beaker. Add 1-3 drops of Methanol or Ethanol.
3. Label the columns on the TLC plate, two standard runs and sample numbers.
4. With a glass cutter, score the plate at the $CaCO_3$ end where the standard and samples are to be added.
5. Clean and rinse syringe with Methanol. Be sure to rinse between each sample also.

6. Add about 1 uL/drop of each standard to the appropriate columns of the TLC plate.
7. Add about 1 uL/drop of each sample to the appropriate columns on the TLC plate.
8. Measure 120 mm from the end of the $CaCO_3$ and mark it with a pencil.
9. Insert the TLC plate into the TLC chamber, cover and allow to elute for about 45-50 minutes or until the solvent reaches the 120 mm pencil mark.
10. Take the plate out of the chamber and mark the line where the solvent stopped with a pencil.
11. Allow plate to dry for 10-20 minutes.
12. When dry, place the plate in the UV Analysis Cabinet
13. Turn on the cabinet and long wavelength (365nm).
14. Read the plate. With a pencil, draw a circle around the LSD standard mark, as well as for the samples.
15. Measure the distance traveled by the LSD. Measure using the center of the spot.
16. Report Rf values for each substance. The Rf value is defined as:

$$Rf = \frac{\text{Distance substance traveled from origin}}{\text{Distance solvent front travels from origin}}$$

17. If sample results are positive, then proceed to Step B, Direct Injection.

B. Direct Injection on GC/MS:

1. This method is done by direct injection on the GC/MS, therefore the standard and blank should be analyzed first before the sample is prepped. However, if the screening procedure was performed then the sample is already prepared so proceed to next step.
2. Direct inject about 1.5 uL of standard. Then direct inject 1 uL of blank. If the sample still needs to be prepped, place the sample in a microvial inserted into an autosampler vial and place 1-3 drops of Ethanol into it, then cap.
3. Direct inject 5-7 uL of the sample.
4. When run is completed and report generated, remove sample (if in paper form) from microvial and return to evidence bag.
5. The GC/FID conditions are:
   Method: LSD.M
   Oven:
   - Initial Temp: 55°C
   - Initial Time: 0.50 min.
   - Max. Temp: 300°C
   - Rate: 60 ^{o}/min.

   Inlet: (front injector only)
   - Mode: Split
   - Initial Temp: 250 ^{o}C

Pressure: 30.0 psi
Gas Type: Helium

Column:

Capillary: 5972: HP-5MS 25m x 200um x 0.33um
5973: HP-1MS 25m x 200um x 0.33um

6. If LSD is present in the sample the instrument will detect a peak at approximately 14.26 minutes on the 5890/5972 and at approximately 13.00 minutes on the 6890/5973. A report will be generates along with accompanying chromatograph and spectra. The spectra will contain of the peak and its ion abundance (See graph, last graph).

V. Results:

A. Record results of GC/MS in logbook and transfer the results to appropriate evidence cards, as well as any paperwork that came with the sample. Be sure to include the date of analysis, result, and initials on the evidence cards.
B. All reports generated from the instrument should be filed so they may be accessed at a later date, if necessary

Section XIV: Identification of Dextromethorphan

I. Introduction:

Samples are routinely analyzed by GC/FID and GC/MS. If the mass spectra results are Dextromethorphan, the sample must be analyzed by a microcrystalline test using 10% Platininc Chloride (H_2PtCl_6) and 1% Acetic Acid. This is done because the GC/MS is unable to distinguish between the optical isomers (dextro and levo) of this particular compound. The microcrystalline test does distinguish between the two.

II. Reagents:

A. 10% Platinic Chloride in water
B. 1% Acetic Acid
C. Dextromethorphan Standard

III. Equipment:

A. Magnifying microscope
B. Microscope slides
C. Microcaps (5uL)

IV. Procedure:

A. Place small amount of sample onto a microscope slide.
B. Add 2 drops of 1% Acetic Acid to the sample.
C. Add 5 uL (1 microcap full) of H_2PtCl_6 to the acetic acid.
D. View at 100X; look for branching root-like crystals.
E. If crystals are present, the sample is either Dextromethorphan OR Levomethorphan. Proceed to Step F. If no crystals form, the sample is a mixture of the two, Racemethorphan.
F. If crystals are resent, repeat steps A through D with the addition of the Dextromethorphan standard to the sample.
G. If crystals form, the sample is Dextromethorphan. If crystals do not form, the sample is Levomethorphan.

V. Results:

A. Record results in logbook, as well as the evidence cards. Be sure to include the date of analysis, result, and initials on the evidence cards.

VI. Discussion:

The microcrystalline test must be performed because the GC/MS cannot distinguish between the Dextro and Levo optical isomers. Since Dextromethorphan is not a controlled substance and Levomethorphan is controlled (Class B), the analyst must be sure which form the sample is before reporting results.

Section XVI: Identification of Anabolic Steroids

I. Introduction:

Suspected Steroid samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. Steroids may either be in the form of tablets or injectable oils.

II. Reagents:

A.) Methylene Chloride (oils dissolve better into this solvent)
B.) Methanol (for extraction and/or solvent rinse for GC.

III. Equipment:

A.) Pipettes
B.) 2 mL autosampler vials with Teflon caps
C.) GC/FID: HP 6890 or 7890 A
D.) GC/MS: HP 6890/5973 or HP 7890A/5975C series.

IV. Procedure:

` A.) Chromatography by GC/FID and GC/MS.

1. Do a visual examination of what the suspected steroid is packaged in, and what it is labeled as. Record in logbook.
2. If in oil form, add 1-2 mL of oil into an autosampler vial and fill to volume with either Methylene Chloride or Methanol.
3. If in tablet form, add half of tablet to an autosampler vial and fill to volume with Methanol.
4. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
5. GC/FID conditions are as follows:
   Method: STER.M
   Oven:
   Initial Temp: 230°C
   Initial Time: 0.00 min.
   Rate: 10°/min.
   Final Temp: 290°C
   Run Time: 10 min.
   Max. Temp: 325°C
   Equilibration Time: 0.5 min.
   Inlet:
   Mode: split (35:1)

   Pressure: 24.99 psi

Gas Type: Helium

Column:

Capillary: HP-1 30m x 320um

Initial Flow: 3.3 mL/min.

Detector:

Temp: 300°C

Hydrogen Flow: 30.0 mL/min.

Air Flow: 400 mL/min.

Makeup Gas: Helium

C.) Obtain chromatographs. If a steroid is present, the instrument will detect a peak and will generate a report with accompanying chromatograph.

D.) Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.

E.) Place same sequence on the GC/MS autosampler and run.

F.) GC/MS conditions are as follows:

Method: STER.M

Oven:

Initial Temp: 230°C

Initial Time: 0.00 min.

Max. Temp: 300°C

Equilibration Time: 0.50 min.

Rate: 10°/min.

Final Temp: 280°C

Run Time: 35 min.

Inlet:

Mode: split (50:1)

Initial Temp: 250°C

Pressure: 18.0 psi

Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um

Max. Temp: 300°C

Initial Flow: 1.0 mL/min.

G.) If an anabolic steroid is present in sample, the instrument will detect a total ion peak and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance (see graph, last page).

V. Results:

A.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section XVIII: Identification of Ketamine

I. Introduction:

Ketamine comes in a clear liquid and a white or off-white powder form. It is also found with other substances such as heroin and MDMA. Samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. Ketamine samples alone requires, by law, a test for the presence of Chloride Ion. This is done by a manual test for chloride ion or by IR.

II. Reagents:

A.) 9:1 Methylene Chloride/Isopropanol or Ethanol
B.) 2M Sulfuric Acid, H_2SO_4
C.) Potassium Persulfate, $K_2S_2O_8$.
D.) .2M Silver Nitrate, $AgNO_3$.

III. Equipment:

A.) Analytical balance
B.) Weigh paper
C.) Pipettes
D.) Glass test tubes
E.) 2 mL autosampler vials with Teflon caps
F.) GC/FID: HP 6890 or 7890A
G.) GC/MS: HP 7890A/5975C or HP 6890/5973 series.
H.) Perkin Elmer Spectrum 100 FT-IR Spectrometer.

IV. Procedure:

` A.) Chromatography by GC/FID and GC/MS.

1. Obtain gross weight of sample evidence bag.
2. Remove one sample packet from the evidence bag and weigh.
3. Record individual packet gross weight in logbook.
4. Remove powder from packet onto a weigh paper.
5. Reweigh the empty packet and record the weight.
6. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
7. Add powder back to packet and reseal.
8. Return packet(s) to original evidence back, reseal, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the powders net weight.

10. For up to 100 packets, analyze 10% of the total. Report the average of the individual the net weights.
11. For more than 100 packets, analyze the square root of the total. Report the average of all the individual net weights.
12. Add 1-2 mL of Ethanol or 9:1 Methylene Chloride/Isopropanol to the autosampler vial(s) containing the sample and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
14. GC/FID conditions are as follows:
    Method: EXP.M
    Oven:
        Initial Temp: 245°C
        Initial Time: 0.00 min.
        Rate: 10°/min.
        Final Temp: 290°C
        Run Time: 10 min.
        Max. Temp: 325°C
        Equilibration Time: 0.5 min.
    Inlet:
        Mode: split (35:1)
        Initial Temp: 250°C
        Pressure: 24.99 psi
        Gas Type: Helium
    Column:
        Capillary: HP-1 30m x 320um
        Initial Flow: 3.3 mL/min.
    Detector:
        Temp: 300°C
        Hydrogen Flow: 30.0 mL/min.
        Air Flow: 400 mL/min.
        Makeup Gas: Helium
15. Obtain chromatographs. If ketamine is present, the instrument will detect a peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
16. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.
17. Place same sequence on the GC/MS autosampler and run.
18. GC/MS conditions are as follows:
    Method: EXP.M

Oven:
Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.
Inlet:
Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium
Column:
Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

19. If ketamine is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

B.) Determining the presence of the Chloride Ion.

1. Analyst may either use the manual Test for Chloride Ion (see below) or the FT-IR Spectrometer.
2. Test for Chloride Ion:
   a. Dissolve 3-5 mg of sample in 1 ml of 2M H_2SO_4. This will eliminate interference from non halides
      i.e. CO_3^{-2}, CN^- and S^{-2} etc.
   b. Add 10-15 mg of $K_2S_2O_8$ .This oxidizes Br^- and I^- but not Cl^- to Br_2 or I_2 . A brown coloration indicates the presence of I2 or Br_2. This will eliminate interference from I^- and Br^-.
   c. Heat to 100° C. for 5-10 minutes. This will drive off halides as gases Br_2 and I_2.
   d. Cool then add 2 Drops of .2M $AgNO_3$. A white precipitate indicates the presence of Chloride (Cl^-). AgF is soluble in water.

V. Results:

A.) Record results of the GC/MS and Chloride Ion Test in logbook. Then transfer the results to appropriate evidence sheets that came with the actual samples. Be sure to include date of analysis, net weight if applicable, number analyzed, results, and signature.

B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section XVII: Identification of Tablets, Capsules and Other Prescription Drugs

I. Introduction:

Class A through E tablets and capsules are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series. The samples are extracted by a simple solvent extraction procedure. However, an extraction procedure specific for non-volatile organic poisons can also be used (See Section IX).

II. Reagents:

A.) Ethanol or Methanol
B.) Methanol (GC solvent rinse)
C.) Petroleum Ether (solvent for certain extractions)
D.) Ammonium Hydroxide, NH_4OH (used for samples containing Acetaminophen)

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2 mL autosampler vials with Teflon caps
D.) Pipettes
E.) GC/FID: HP 6890 or 7890A series
F.) GC/MS: HP 7890A/5975C or HP 6890/5973 series
G.) Computer with Identidex Imprint Identification program and/or online resources such as drugs.com, pharmer.org.
H.) PDR reference books and DrugBible textbook.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the computer's description matches your sample's description.
7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. All Class A through D tablet, capsule and other prescription drugs must be analyzed by GC/FID and GC/MS.
2. Most Class E prescription drugs are analyzed by visual description only except for Steroids (See Section XVI)
3. If capsule or tablet contains no identifiable imprint code or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
4. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
5. Add 1-2 mL of Ethanol or Methanol, or 1 drop of NH_4OH and 1-2 mL of Petroleum Ether if the sample contains Acetaminophen.
6. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples).
7. GC/FID and GC/MS conditions are as follows:
   Methods: EXP.M, EXPSL.M, STER.M
   Oven:
      Initial Temp: 245°C
      Initial Time: 0.00 min.
      Rate: 10°/min.
      Final Temp: 290°C
      Run Time: 10 min, 15 min, 30 min, respectively
      Max. Temp: 325°C
      Equilibration Time: 0.5 min.
   Inlet:
      Mode: split (35:1)
      Initial Temp: 250°C
      Pressure: 24.99 psi
      Gas Type: Helium
   Column:
      Capillary: HP-1 30m x 320um
      Initial Flow: 3.3 mL/min.
   Detector:
      Temp: 300°C
      Hydrogen Flow: 30.0 mL/min.
      Air Flow: 400 mL/min.
      Makeup Gas: Helium

   Method: HYD.M
   Oven:
      Initial Temp: 130°C

Initial Time: 2.50 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 30 min.

Inlet:

Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium

Column:

Capillary: HP-1MS 30m x 0.530mm
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

8. Obtain chromatographs. If sample contains any controlled substance the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph and spectra (MS). The spectra will contain the identity if the peak and its ion abundance.
9. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased. Also observe any erroneous data that indicates that the sample may have to be reinjected.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook, as well as on the evidence cards that came with the samples. Be sure to include date of analysis, results, and initials. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence sheets that came with the actual samples. Be sure to include date of analysis, net weight if applicable, number analyzed, check whether analyzed by appearance and labeling only, results, and signature.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Section XV: Identification of Hallucinogenic Mushrooms

I. Introduction:

Peyote Buttons are first given a visual test to check for their characteristic. The peyote buttons are the top of the cactus, only about two inches in diameter and ½ inch tall, they are rounded and grayish-green in color, there are no spines on the cactus but it does have short tuft of hair growing on top.. Then the peyote sample are solvent extracted using Ethyl Ether. This extract is then run on the GC/MS. If the peyote sample is positive the active ingredient Mescaline will be detected on the GC/MS.

II. Reagents:

A.) 3 N Acetic Acid (HOAC): 37.5 mL glacial acetic acid in 412.5 mL distilled water.

B.) Ethyl Ether
C.) Ammonium Hydroxide (NH_4OH).
D.) Methanol

III. Equipment:

A.) 250 mL glass beakers (4).
B.) Mortar and pestle.
C.) 7.0 cm filter paper.
D.) 250 mL volumetric vacuum flask.
E.) Glass separatory funnels with stoppers and stopcocks (2).
F.) Glass stir rod.
G.) pH paper.
H.) Glass pipettes.
I.) Hot sand bath.
J.) 5 uL autosampler syringe (for manual injections)
K.) GC/MS: HP 6890/5973 or HP 7890A/5975C series

IV. Procedure:

A.) Extraction Using Ethyl Ether:

1. Observe peyote buttons or shavings and compare to literature.
2. Dry peyote buttons at 100 degrees for ½ hour.
3. Weigh out 2.5 g of sample in a tared 250 mL beaker. Be sure to include all parts of the button.
4. Grind the sample into finer pieces with a mortar and pestle. Add back to the beaker.
5. Fill beaker with 3 N HOAC so solution is just above the sample and so sample is saturated. Let sit overnight in a dark place (ex. drawer).
6. Vacuum filter with 3 N HOAC, making sure to rinse the beaker very well.
7. In a hood, extract with ethyl ether by pouring the filtered sample into a glass separatory funnel, add ethyl ether (5:1 organic to aqueous), stopper and invert a several times. Make sure to vent the funnel to let excess gas inside the funnel escape.
8. Remove stopper and allow the layers to separate. The ethyl ether layer will be on the top.
9. Drain the bottom layer directly into another glass separatory funnel and discard the ethyl ether layer into appropriate waste container.
10. Add NH_4OH until solution is basic.
11. Extract again with ethyl ether (5:1 organic to aqueous). This time discard the bottom layer into waste container and drain the ethyl ether layer in to a beaker labeled with the sample number.
12. Either let the sample evaporate overnight in a dark place, or place on a hot sand bath until dry.

13. Rinse the beaker with Methanol, swirling to make sure the sides of the beaker get hit with Methanol.

B.) Chromatography by GC/MS:

1. Manually inject, in the following order, 1.5 uL of the mescaline standard, blank, and sample(s) directly from the beaker into the GC/MS.
2. GC/MS conditions:
   Method: HYD.M
   Oven:
   - Equilibration Time: 0.50 min.
   - Initial Temp: 135°C
   - Oven Max: 325°C.
   - Rate: 10°/min.
   - Final Temp: 280°C
   - Run Time: 30 min.

   Inlet:
   - Mode: split (50:1)
   - Initial Temp: 250°C
   - Pressure: 31.65 psi
   - Gas Type: Helium

   Column:
   - Capillary: HP-1MS 25m x 200um x 0.33um
   - Max. Temp: 300°C
   - Initial Flow: 1.0 mL/min.
3. If mescaline is present in sample, the instrument will detect a total ion peak at its retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance (see graph, last page).

V. Results:

A.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.

Test for Chloride Ion

1. Dissolve 3-5 mg of sample in 1 ml of 2M H_2SO_4. This will eliminate interference from non halides i.e.
CO_3^{-2}, CN^- and S^{-2} etc.

2. Add 10-15 mg of $K_2S_2O_8$ .This oxidizes Br^- and I^- but not Cl^- to Br_2 or I_2 . A brown coloration indicates the presence of I2 or Br_2. This will eliminate interference from I^- and Br^-.

3. Heat to 100° C. for 5-10 minutes. This will drive off halides as gases Br_2 and I_2.

4. Cool then add 2 Drops of .2M $AgNO_3$. A white precipitate indicates the presence of Chloride (Cl^-). AgF is soluble in water.

Reagents:

.2M AgNO3	340mg/10mls
.2M KCl	150mg/10mls
.2M KBr	238mg/10mls
.2M KI	330mg/10mls
2M H2SO4	11mls/100mls