Controlled Substances
Training Manual

1.0 Purpose

The following manual is to be used as a guide in training new criminalists in the analysis of controlled substances. It may be used in its entirety or for just the sections of interest. Each section is to be used with the corresponding SOP found in the ISP Controlled Substances SOP manual. Special thanks are given the Kansas Bureau of Investigations for their permission to use their manual as the basis for this manual.

2.0 Procedure

As each subsection is completed both the criminalist and the trainer will sign and date the checklist. Once all training is complete then the analyst will get a copy of the checklist and the original will go to the QA/QC manager to be kept with the analyst's other training records.

3.0 General Laboratory

3.1 Safety Manual
Read safety manual.

3.2 Quality Manual
Read quality manual.

3.3 Evidence Procedures
Review evidence procedures including, but not limited to, evidence receipt and checkout, proper seals, and the IETS system

3.4 Demonstrated competency of theory and use of the various balances in the laboratory including but not limited to the operation of tare features and the monthly calibration check. Read section 28B, pg 533 through 540, of *Analytical Chemistry, by Skoog/West/Holler, 6th edition.*

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4.0 Marijuana

4.1 Background reading
To include Idaho Code sections 37-2701 (n)(s) and 37-2707 d(27),
"Drug Identification Bible, 2002". Pages 617-665.
Worksheets and reporting format.
MJ training.doc
TLC training.doc

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Trainer

Sections 4.2, 4.3, 4.4 and 4.5 are used with the Marijuana SOP.

4.2 Physical examination, SOP section 5.0.0
4.2.1 Microscopic examination of cystolithic and unicellular hairs, seed structure.
4.2.2 Seed germination, SOP section 9.0.0

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4.3 Duquenois Levine
SOP section 7.0.0.

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4.4 Thin Layer Chromatography (TLC)
4.4.1 Basic theory and SOP section 6.0.0
4.4.2 Chloroform and Pet ether/ ether systems with fast blue BB.

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4.5 Hashish and Pipes
Order of analysis and reporting differences.

Date of completion _____ Criminalist

Trainer

4.6 Competency test

100 % correct analysis of various plant, and other, material. The competency test will be provided by the discipline leader.

Date of completion _____ Criminalist

Trainer

4.7 Mock Court

Purpose, layout and critique.

Date of completion _____ Criminalist

Trainer

4.8 Cosigned Case Review

One hundred (100) and letter from trainer.

Date of completion _____ Criminalist

Trainer

5.0 Solid Dosage Drugs

5.1 Background reading

The Idaho Code, all of section 37-2700.

Drug Identification Bible, 2000. Applicable sections.

Date of completion _____ Criminalist

Trainer

5.2 Gas Chromatograph Mass Spectrometer (GC/MS)
5.2.1 Hewlett Packard GC/MS tutorial
5.2.2 Review of maintenance procedures and schedules
5.2.3 Review of documentation and filing system
5.2.4 HP Chemstation software
5.2.5 Review of various temperature programs
5.2.6 Read GC training.doc and MS training.doc
5.2.7 GC/MS SOP.

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5.3 Fourier Transform Infrared Spectrometer
5.3.1 Nicolet tutorial
5.3.2 Nicolet Omni software
5.3.3 Review maintenance procedures, schedules, and documentation
5.3.4 Spectroscopy training.doc and IR training.doc

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Trainer

5.4 Chemical color (spot) tests
5.4.1 Review Clark
5.4.2 Spot tests training.doc
5.4.3 Spot tests of known standards with various reagents.

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Trainer

5.5 Phenethylamine family

5.5.1 Review Phenethylamine SOP.

5.5.2 Review CTMAMPH training.doc

Date of completion _____ Criminalist

Trainer

5.6 Cocaine family

5.6.1 Read Cocaine SOP.

5.6.2 Review Cocaine training.doc

Date of completion _____ Criminalist

Trainer

5.7 Opiates

5.7.1 Review Opiates SOP.

Date of completion _____ Criminalist

Trainer

5.8 Mushrooms

5.8.1 Review Psilocyn/Psilocybin SOP

5.8.2 Review CTMHAL.doc

5.8.3 TLC analysis with T1

Date of completion _____ Criminalist

Trainer

5.9 Pills, LSD, and general unknowns

5.9.1 Review references i.e. PDR, Logo, etc. DEA and Idaho code scheduling
5.9.2 Review Extractions.doc and CTMHAL training.doc
Date of completion _____ Criminalist

Trainer

5.10 Clandestine Laboratory Analysis (common)
5.10.1 Review analytical selection sequence.
5.10.2 Phosphorous
5.10.2.1 Sigma Kit
5.10.2.2 Other chemical and physical methods
5.10.3 Iodine chemical and physical methods
5.10.4 Pseudoephedrine and ephedrine extractions and analytical procedures
5.10.5 Methamphetamine extraction and analysis

Date of completion _____ Criminalist

Trainer

5.11 Competency test
100% correct identification of controlled substances from a variety of powders, liquids, and or pills. Results will be attained using all aspects of analysis including at least one result from each instrument capable of producing structural information, i.e. MS and FTIR. The competency test will be provided by the discipline leader.

Date of completion _____ Criminalist

Trainer

5.12 Mock court
Purpose, layout, and critique

Date of completion _____ Criminalist

Trainer

5.13 Cosigned casework review

One hundred (100) and letter from trainer

Date of completion _____ Criminalist

Trainer

#15
Standard Operating Procedures For the Analysis of Liquid Pharmaceutical Samples Using GC/MS

1.0.0 Background

Under normal circumstances quantification of a substance's purity is not part of the analytical scheme used by the Idaho State Police Forensic laboratories. By special request this analysis can be performed. Cases involving suspected tampering of liquid pharmaceuticals are analyzed using this method. The basis for this method is the comparison of peak areas between a reference standard of known concentration vs. samples. By comparing the peak areas and utilizing a simple ratio, one can determine if a sample has been altered. The purpose of this method is not to determine the exact concentration of the samples; rather it is to determine whether a sample has been altered at all. The sample and the reference standard must be prepared using the same technique.

2.0.0 Scope

Although the following procedure has only been tested using Demerol (meperidine), the principal behind it is sound and will allow for the analysis of other injectable pharmaceuticals as long as the appropriate reference standards and solvents are used.

3.0.0 Equipment and Reagents

3.1.0 Gas Chromatograph/ Mass Spectrometer (GC/MS) and corresponding software.
3.2.0 Reference standard provided by Board of Pharmacy. Must be of the same listed concentration as the samples.
3.3.0 GC or pesticide grade chloroform, or other appropriate solvent.
3.4.0 Volumetric flasks (5 and 10ml).
3.5.0 Gas tight syringes in a variety of sizes. (2.5ml, 250ul, and 25ul work well)

4.0.0 Sample and Standard Preparation

Typically the concentration of injectable pharmaceuticals is such that they need to be diluted b[illegible] formed in order not to overload the GC column or the MS dete[illegible]

4.1.0 [illegible] greater than 10mg/ml then dilute the [illegible]1 using gastight syringes.
4.2.0 [illegible] add 500ul of solvent and any base or [illegible]d shake.
4.3.0 [illegible].ce in a sample vial equipped with an
4.4.0
4.5.0 [illegible]dard and sample(s), calculate result.

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7/03 - stopped use
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5.0.0 Calculation of Final Results

Using a simple ratio of areas calculate the relative response:

$$\frac{\text{(Area of sample) X 100}}{\text{(Area of Standard)}} = \%\ \text{sample relative to standard}$$

6.0.0 Conclusions

The results are to be reported as a relative percent of the standard provided by the Board of Pharmacy.

7.0.0 Notes and QA/QC

7.1.0 Injector should have a split liner with a glass wool plug.

7.2.0 It is acceptable to use either manual or instrument generated integration. The analyst must be consistent however in how the integration is applied if using a manual method.

7.3.0 The reporting limit is (+/-) 15% (of calculated response).

#9
Opiates
Standard Operating Procedures

1.0.0 Background

Opium is the resin from the plant *Papaver Somniferum*. It has been used for medicinal purposes for at least 6000 years. Typically good quality opium will contain ten to fourteen percent morphine. Another natural opiate found in opium is codeine. Diacetylmorphine (Heroin), as the chemical name implies is a modified morphine product. All the opiates are listed as controlled substances.
Further information about opiates can be found in;
"Drug Identification Bible", 2002 Edition.
"Methods of Analysis", IRS publication 341 (Rev 6-67)

2.0.0 Scope

The following analytical procedures are used to confirm the presence of opiates in samples. Normally when analyzing a sample submitted as suspected heroin, only the diacetylmorphine will be confirmed.

3.0.0 Equipment and Reagents

The following pieces of equipment can be used to identi
3.1.0 A GC/MS and appropriate analy
3.2.0 FTIR and appropriate analytical
3.3.0 Reagent grade solvent. Chlorofo nay be used.

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obsolete 1/12/07
No new revision

4.0.0 Color Spot Tests

Marquis, Mecke's, and Froehde's are som
screen for the presence of opiates.
Recipes for these reagents can be found in
Drugs", 2nd Edition, 1986.

5.0.0 GC/MS Sample Preparation and Analysis

5.1.0 Sample preparation (Heroin).
5.1.1 Samples and standards are extr
5.1.2 Samples and standards are diss ..oak acid, and then made basic with NaHCO3. The solution is extracted using an immiscible solvent.
5.2.0 Other forms of opiates
By far the most prevalent, non-heroin, opiates are found in pills and to a lesser degree in injectable pharmaceuticals. To analyze these samples see the Unknown

SOP.

5.3.0 GC/MS analysis. The retention time of the sample should be within 0.04 minutes of a valid MS scan from the standard. Refer to Gendrug and or GC/MS SOP for a definition of a valid scan.

6.0.0 FTIR Sample Preparation Methods

Heroin "Panning" technique from NWAFS newsletter Vol. 12 No. 1.

6.1.0 Dissolve 100mg, preferably more, of sample (Black tar) in 5 mls of 10% HCl. Filter through cotton or glass wool if necessary.

6.2.0 Extract with 5mls of chloroform. Discard the aqueous, acidic, layer.

6.3.0 Back extract with 5 mls of water. Save aqueous layer. Repeat, combining both aqueous layers. Discard chloroform.

6.4.0 Add sodium bicarbonate and extract three times with 5 mls of chloroform. NOTE at this time the solvent can be evaporated onto KBr and analyzed to yield heroin base. May not yield good results due to the polymorphic nature of the base and the base is sticky and it may be difficult to press a good KBr pellet.

6.5.0 Bubble HCl through the chloroform. Dry through a sodium sulfate column. May be analyzed at this stage by drying on KBr.

6.6.0 Using an air stream and heat, recrystalize by doing a solvent exchange with petroleum ether. Evaporate onto KBr.

6.7.0 The spectra of the sample must be compared to a spectra of a standard that was prepared using the same procedure.

#7
Phenethylamine Family
Standard Operating Procedures

1.0.0 Background

The phenethylamine family of drugs include, but are not limited to, amphetamine, methamphetamine, phentermine, ephedrine, pseudoephedrine, 3,4-MDA, and MDMA. Almost all are controlled substances, or can be used to make controlled substances, and can be identified by several different techniques currently used by the ISP-Forensic Service laboratories. General information can be found in numerous articles (Microgram etc.) and books such as "Drug Identification Bible", 2002 edition.

2.0.0 Scope

The following analytical procedures are used to con[illegible]sence of phenethylamines in samples.

3.0.0 Equipment and Reagents

The following pieces of equipment [illegible]dentify the analytes of interest.

3.1.0 A GC/MS and appr[illegible]OP.

3.2.0 FTIR and appro[illegible]

3.3.0 Polarizing micros[illegible]

4.0.0 Color Spot Tests

Marquis, Liebermanns, and so[illegible]st common spot tests used to help i[illegible], and other reagents can be found in "Cla[illegible]gs" 2nd Edition, 1986.

5.0.0 GC/MS Sample Preparation and Analy[illegible]

Either of two extraction methods can be us[illegible]ne analyst's discretion.

5.1.1 Basic Extraction. Place approximately [illegible]ample into a test tube. Dissolve with distilled water. Make basi[illegible]n Na2CO3 or another strong base. Extract with petroleum ether, hexane, or other non-water soluble solvent. Analyze on GC/MS. The standards are prepared using this method. **NOTE** Amphetamine and methamphetamine basic extracts are volatile. If the extract in the sample vial is allowed to completely evaporate then the analyte may be lost. It is important to recap the sample vial with a new septa if the extract needs to be saved for reanalysis or returned to evidence in a trace case where all of the original sample was used.

5.1.2 Direct extraction. A small amount of the sample, approximately 0.01 g, is dissolved in methanol or other appropriate solvent, and analyzed on the GC/MS.

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obsolete 1/12/07
No new revision

5.2.0 GC/MS analysis. The retention time of the sample should be within 0.04 min of a valid MS scan from the daily standard. **NOTE** Underivatized ephedrine and pseudoephedrine cannot be separated using normal GC/MS columns. To identify either separately, the sample must be analyzed using a FTIR.

6.0.0 FTIR Sample Preparation Methods

When purification is necessary the following methods should be used in making KBr pellets. Alternative sample introduction techniques can be used when appropriate.

6.1.0 Amphetamine and Methamphetamine.

6.1.1 Dissolve sample in water or dilute acid. Make basic with Na2CO3, or another strong base, and extract with petroleum ether or hexane. Wash extract with water then dry through Na2SO4. Bubble HCl gas through solvent and collect the resulting crystals. Wash with additional petroleum ether and let dry. Make KBr pellet. Compare against a known HCl salt standard.

6.1.2 dl-Methamphetamine by PIT derivative.
Dissolve sample in water and make basic with Na2CO3. Extract with petroleum ether and dry extract through NaSO4. Add 2 drops of phenylisothiocyanate(PIT) and let stand for 10 minutes. Decant solvent and wash crystals with additional solvent. Dry and make a KBr pellet. Compare with a dl-Methamphetamine standard that was prepared using this procedure.

6.1.3 Direct. Mix and grind sample with KBr. Form into a pellet.

6.2.0 Ephedrine and Pseudoephedrine.

6.2.1 Using the same procedure as 6.1.1 will yield the HCl salt.

6.2.2 To obtain the base form, dissolve the sample in water, make basic and extract with petroleum ether or hexane. Evaporate the solvent and make a KBr pellet. Compare against a known base standard.

6.2.3 If the sample is a pill(s), crush and add methanol and shake well. Wait approximately one hour and centrifuge. Place supernatant into a clean test tube and allow too evaporate. Take crystals from side of test tube and make a KBr pellet.

#12

General Unknowns
Standard Operating Procedures

1.0.0 Background and Scope

There are times when samples do not fit into a certain category. These procedures are designed to analyze these samples, examples of, which are pills, liquid pharmaceuticals, and samples that do not give the expected results with screening tests. Whenever possible, two different tests, and two different sampling events will be employed in confirming the presence of controlled substances. One of the tests must provide structural information, i.e. either MS or FTIR.

2.0.0 Equipment and Reagents

The following pieces of equipment can be used in ... analytes of interest.

2.1.0 A GC/MS and appropriate analyti...

2.2.0 FTIR and appropriate analytical s...

2.3.0 Reagent, or better, grade solvents.

3.0.0 GC/MS Sample Preparation and Analysi...

3.1.0 Extraction.

3.1.1 Pills. Using appropriate samp... other solvent.

3.1.2 Powders. Extract with methan... solvent.

3.1.3 Aqueous samples, including inj... chloroform or other appropriate ...

3.1.4 Methanol washes can be injected ...

3.1.5 Acidic or basic shakeouts can be ... other to separate diluents or other interferences. These A/B extractions can be performed with commercial products (Toxi-Lab tubes) or laboratory generated solutions.

3.2.0 Analysis.

3.2.1 Run samples using a general unknown data acquisition method.

3.2.2 If a peak appears, perform a library search.

3.2.3 If a controlled substance is recognized from a library search or other means, then a standard is run if identity is to be confirmed. Library search reports do not need to be retained in the case file.

3.3.0 Conclusions.

3.3.1 Confirmation. The retention time must be within 0.04 min of a valid scan of the standard and the MS spectra must match. If both conditions are satisfied then confirmation can be reported.

3.3.2 Non-confirmation. If a standard is not available but the library search produces a match then report the presence of the compound with a "not

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confirmed" statement.

3.3.3 If the RT or MS do not match, or there is no peak at all, then report, "No controlled substances detected".

3.3.4 As with all cases it is up to the analyst to decide whether or not to report non-controlled substances.

4.0.0 FTIR Sample Preparation and Analysis Methods

4.1.0 Direct.

Powders may be ground with KBr and run directly or analyzed using the microscope attachment. Organic liquid samples may be placed on a window of KBr or a commercial equivalent.

4.2.0 Extractions

4.2.1 The organic layer of the above extractions may be mixed with ground KBr, evaporated and analyzed (do not use methanol or other solvent containing water).

4.2.2 Samples undergoing a basic extraction may require bubbling with HCl gas and filtering before HCl salt can be isolated and analyzed.

4.3.0 Analysis

4.3.1 Analyze samples per FTIR SOP.

4.3.2 Perform a library or literature search of the resulting spectra.

4.4.0 Conclusions

4.4.1 Confirmation.

If the spectra of the standard in the ISP Forensics produced library and sample match in all significant respects the compound may be reported.

4.4.2 Non-confirmation

If a spectra from an ISP Forensics produced library is not available but the library or literature search produces a match the presence of the compound may be reported with a "not confirmed" statement.

4.4.3 If no spectral match is found report the sample as "No controlled substances detected".

4.4.4 The analyst may decide whether or not to report non-controlled substances.

#14
Phosphorus
Standard Operating Procedures

1.0.0 Background

The following set of procedures are used to identify elemental phosphorus. Phosphorus is typically found at clandestine methamphetamine laboratories. Although white phosphorus may be found, red phosphorus is most often encountered due in large part to its greater accessibility.

2.0.0 Scope

The following analytical procedures are used to identify the presence of phosphorus. The tests are listed in decreasing order of specificity. The GC/MS procedure is the only one that can be used, by itself, to confirm the presence of phosphorus. The "Sigma" kit has two possible interferences and thus must be used in combination with some other test in order to identify phosphorus. The other tests are considered screening tests.

3.0.0 GC/MS Confirmation

3.1.0 Equipment and reagents

- 3.1.1 A GC/MS and appropriate analytical software. Reference GC/MS SOP.
- 3.1.2 Test tube and holder, Bunsen burner
- 3.1.3 Chloroform, reagent grade.

3.2.0 Procedure

This test is based on the fact tha[t] [ ] while red phosphorus is not. Being so [ ] nto a GC/MS.

- 3.2.2 Conversion of red to whi[te] phosphorus into a test tub[e] phosphorus starts to emit y[ellow] Remove from heat and imm[ediately] is present then the above ste[p] will boil and spit when added[.] ignite upon exposure to air. T[his] hood.
- 3.2.3 Analyze the extract on the GC/[MS] presence of the P2, P3, P4 ions [at] respectively) and compared to a [standard]

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4.0.0 "Sigma" Kit

The Sigma 670 kit uses the principal that inorganic phosphorus will form a blue color

complex when reacted with an acidic molybdate solution and then reduced.

4.1.0 Follow the instructions provided with the kit excluding the spectrophotometer since we do not care about concentration. The presence of phosphorus is determined when the blue color is observed. **NOTE.** The red phosphorus found at clan labs most often is recovered from matchbooks. Some of the glue can be mixed in with the sample and it may take up to thirty minutes for the blue color to develop.

4.2.0 Interferences
Arsenic and silica (quartz) give false positives with this test.

5.0.0 Screening Tests

5.1.0 pH shift.
Phosphorus, when added to iodine, will form hydriotic acid in an aqueous solution thus lowering the pH of the solution.

5.1.1 Add the sample to water and measure pH. If the solution is acidic then wash with water until it is neutral. Once the sample is neutral, add some elemental iodine. Check for an acidic pH shift indicating the formation of hydriotic acid. Run an iodine/water control.

5.2.0 Scratch test

5.2.1 Smear a small amount of dried sample on a piece of silicon carbide sandpaper or other similar rough surface. Strike the smear with a safety match. If the match ignites then it is considered a positive test.

5.2.2 Cautions: Too much phosphorus may ignite the paper as well as the match.

5.3.0 Bang test

5.3.1 Place small amount of sample into a mortar and cover with methanol. Add equal amount (to sample) of potassium chlorate ($KClO_3$). Grind and mix with pestle. Do not let the slurry dry out while grinding. Pour slurry into folded bindle of weigh paper and let dry. Hit with a hammer.

5.3.2 Cautions: This test produces and detonates a contact explosive, thus hearing and eye protection is necessary.

6.0.0 Conclusions

6.1.0 The GC/MS procedure by itself can give a positive confirmation.

6.2.0 The Sigma kit, plus one of the screening tests, can give a positive confirmation.

6.3.0 The Sigma kit alone, or any of the screening tests, can only give a presumptive result.

Iodine
Standard Operating Procedures

1.0 Background and Scope

Iodine is one of the essential ingredients in the production of methamphetamine using the ephedrine reduction method. The following methods, when used in combination, can be used to confirm the presence of iodine in samples typically found at clandestine laboratories. The primary test is the Leuco Crystal Violet method. By itself this test cannot be used to confirm the presence of iodine due to the possible false positive reaction to oxides of manganese (MnO2 and MnO4). By using any of the additional tests the presence of the interferences is eliminated thus confirming iodine. A full copy of the Leuco Crystal Violet method can be found in Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-I B.

2.0.0 Equipment and Reagents for Leuco Crystal Violet

2.1.0 Equipment.

- 2.1.1 Flasks and stoppers.
- 2.1.2 Pipettors or glass pipets.

2.2.0 Reagents. All reagent solutions can be scaled up or down from the following recipes.

- 2.2.1 Stock iodine standard. Dissolve 1.3081g of KI into 1 L of distilled water.
- 2.2.2 Citric Buffer.
  - 2.2.2.1 Citric acid. Dissolve 192.2g C6H8O7 or 210.2g C6H8O7.H20 into 1 liter of water.
  - 2.2.2.2 Ammonium hydroxid [illegible] to 1L with water.
  - 2.2.2.3 Final Buffer. Mix 35 [illegible] 0g ammonium dihydro
- 2.2.3 Leuco crystal violet indicat [illegible]
  - 2.2.3.1 Add 200ml water, [illegible] tris (N,N-dimethylanil [illegible] each addition. **
  - 2.2.3.2 Dissolve 2.5g me [illegible]
  - 2.2.3.3 Add HgCl2 solut [illegible] s than 1.5 with H2 [illegible] way from light.
- 2.2.4 Oxone. Potassium [illegible] into 1L of water.

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3.0.0 Procedure for Leuco Crystal Violet

Since our goal in using this test is only the identification of iodine and not the quantification the following procedure has been shortened from the original. The following recipe is based on a final volume of 100 mls for both standard and sample solutions. Using the same proportion of reagents the volume can be successfully reduced. A test is considered positive if a violet color is developed. As with all procedures a blank and a standard are run with every batch.

3.1.0 Standard and blank preparation. Add 0.25 mls of iodine standard to a 100 ml flask. Dilute with 50 to 75 mls of water. Add 1ml citric buffer and 0.5ml KHSO5 solution. Mix and let stand one minute. Add 1ml Leuco violet indicator, mix, and dilute to 100 mls. Color will often develop immediately. If not wait up to five minutes.

3.2.0 Sample Preparation. The most difficult part of this analysis is judging how much sample to use. It is easy to use too much sample. If this happens, a light blue-green- yellow color will develop instead of the expected violet.

3.2.1 Solids. Place a small piece of sample in a volumetric flask. Dissolve with 50 ml of water. Wait approximately one minute. Sample does not need to be completely dissolved. Proceed as with the standard.

3.2.2 Liquids. Place a small amount of sample into a volumetric flask. Dilute with 50 to 75ml of water. The color of the sample solution at this point should be a very light yellow. Proceed as with the standard.

4.0.0 Complementary Methods for the Detection of Iodine

4.1.0 Heat. When a capped test tube containing solid iodine is heated, violet fumes are created. Condensation of the fumes into shinny grey crystals at the cool top of the test tube will often be observed. The oxides of manganese that interfere with the Leuco crystal violet method do not produce this effect.

4.2.0 Starch Test. A liquid iodine solution when added to starch paper produces a blue-black stain, solutions made from the oxides of manganese do not.

4.3.0 Hexane Color Test. Hexane turns to a violet color when added to an iodine solution. Once again solutions containing oxides of manganese do not produce this effect.

4.4.0 pH Shift. Add sample to water and check pH, it should be neutral. Add red phosphorus and let stand. Check for a drop in pH (<2).

History for the Iodine SOP

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux

Approval

Technical Leader _____ Date: 3-28-01
David Sincerbeaux

QA/QC Manager _____ Date: 3-29-01
Rick Groff

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Iodine
Standard Operating Procedures

1.0 Background and Scope

Iodine is one of the essential ingredients in the production of meth- ...using the ephedrine reduction method. The following methods, whe... ...can be used to confirm the presence of iodine in samples f... laboratories. The primary test is the Leuco C... cannot be used to confirm the presenc... reaction to oxides of manganese (MnC... ...sts the presence of the interferences is elim... Leuco Crystal Violet method can be foun... Water and Wastewater, 20th Edition, 1998...

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2.0.0 Equipment and Reagents for Leuco Crys...

2.1.0 Equipment.

2.1.1 Flasks and stoppers.

2.1.2 Pipettors or glass pipets.

2.2.0 Reagents. All reagent solutions can be sca... ...ollowing recipes.

2.2.1 Stock Iodine standard. Dissolve 1.30... ...n into 1 L of distilled water.

2.2.2 Citric Buffer.

2.2.2.1 Citric acid. Dissolve 192.2g C6H8O7 or 210.2g C6H8O7.H20 into 1 liter of water.

2.2.2.2 Ammonium hydroxide 2N. Dilute 131ml of conc. NH4OH to 1L with water.

2.2.2.3 Final Buffer. Mix 350ml 2N NH4OH to 670ml citric acid. Add 80g ammonium dihydrogen phosphate (NH4H2PO4) stir to dissolve.

2.2.3 Leuco crystal violet indicator.

2.2.3.1 Add 200ml water, 3.2ml H2SO4, and 1.5g 4,4',4'=-methylidynetris (N,N-dimethylaniline) ** to a 1L brown glass bottle. Mix upon each addition. ** NOTE** AKA Leuco crystal violet.

2.2.3.2 Dissolve 2.5g mercuric chloride (HgCl2) into 800ml water.

2.2.3.3 Add HgCl2 solution to Leuco crystal violet. Adjust pH too less than 1.5 with H2SO4 if necessary. Store for up to 6 months, away from light.

2.2.4 Oxone. Potassium peroxymonosulfate (KHSO5). Dissolve 1.5 g into 1L of water.

3.0.0 Procedure for Leuco Crystal Violet

Since our goal in using this test is only the identification of iodine and not the quantification the following procedure has been shortened from the original. The following recipe is based on a final volume of 100 mls for both standard and sample solutions. Using the same proportion of reagents the volume can be successfully reduced. A test is considered positive if a violet color is developed. As with all procedures a blank and a standard are run with every batch.

3.1.0 Standard and blank preparation. Add 0.25 mls of iodine standard to a 100 ml flask. Dilute with 50 to 75 mls of water. Add 1ml citric buffer and 0.5ml KHSO5 solution. Mix and let stand one minute. Add 1ml Leuco violet indicator, mix, and dilute to 100 mls. Color will often develop immediately. If not wait up to five minutes.

3.2.0 Sample Preparation. The most difficult part of this analysis is judging how much sample to use. It is easy to use too much sample. If this happens, a light blue-green- yellow color will develop instead of the expected violet.

3.2.1 Solids. Place a small piece of sample in a volumetric flask. Dissolve with 50 ml of water. Wait approximately one minute. Sample does not need to be completely dissolved. Proceed as with the standard.

3.2.2 Liquids. Place a small amount of sample into a volumetric flask. Dilute with 50 to 75ml of water. The color of the sample solution at this point should be a very light yellow. Proceed as with the standard.

4.0.0 Complementary Methods for the Detection of Iodine

4.1.0 Heat. When a capped test tube containing solid iodine is heated, violet fumes are created. Condensation of the fumes into shinny grey crystals at the cool top of the test tube will often be observed. The oxides of manganese that interfere with the Leuco crystal violet method do not produce this effect.

4.2.0 Starch Test. A liquid iodine solution when added to starch paper produces a blue-black stain, solutions made from the oxides of manganese do not.

4.3.0 Hexane Color Test. Hexane turns to a violet color when added to an iodine solution. Once again solutions containing oxides of manganese do not produce this effect.

4.4.0 pH Shift. Add sample to water and check pH, it should be neutral. Add red phosphorus and let stand. Check for a drop in pH (<2).

#

GHB, GBL, and 1,4 BD
Standard Operating Procedures

1.0.0 Background

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

The following analytical scheme was developed to separate and identify GHB, GBL, and 1,4 BD while ensuring that GHB is not produced during the process.

2.0.0 Scope

The following analytical procedures a its related analogs in samples.

GHB Rev 0 9/03 replaced 1/12/07

3.0.0 Equipment and Reagents

The following pieces of equipment cal

3.1.0 A GC/MS and appropriate anal

3.2.0 FTIR and appropriate analytica

3.3.0 Reagent grade chloroform, ethy) a TMS derivatizing reagent. *NO Cerilliant Corporation.

4.0.0 Screening Tests

4.1.0 Color Spot Test

A mixture of Bromocresol green, Methy reagents are tested with samples. A positive reaction for the presence of GHB is one that turns green.

4.1.1 Bromocresol green
Mix 0.03g bromocresol green in 100 mL of 4:1 methanol: water. Adjust to pH 7.0 with NaOH.

4.1.2 Methyl Orange
Mix 0.01g of methyl orange in 100 mL of methanol. Adjust pH to 7.0 .

4.1.3 Modified Schweppes
Solution A: mix 2.0g dextrose in 20 mL of water.
Solution B: mix 2.4g aniline hydrochloride in 20 mL of ethanol.
Mix solution A & B and dilute to 80 mL with methanol.

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

4.2.0 Physical tests.

4.2.1 Pure GBL and 1,4 BD are viscose liquids at room temperature. 1,4-BD will solidify when placed in a refrigerator (4C) while GBL will not.

4.2.2 GBL is soluble in chloroform and 1,4 BD is not.

4.3.0 GC/MS

Add concentrated Sulfuric acid to aqueous sample, extract with chloroform and analyze. If GBL is detected then proceed with confirmational GC/MS.

Recipes for other reagents can be found in "Microgram, Vol XXXV, No.1 January 2002".

5.0.0 GC/MS Sample Preparation and Analysis

GHB cannot be analyzed directly on a GC/MS as it will convert to GBL in the heated injector port. GHB must be derivatized with BSTFA before injection.

5.1.0 1,4-Butandiol

5.1.1 If pure 1,4 BD is suspected then dilute with methanol and inject into GC/MS.

5.1.2 In aqueous samples, if the concentrations of 1,4 BD are high enough, then the 1,4 BD may be observed in a chloroform extract.

5.1.3 Dry down sample, add methanol, and analyze.

5.1.4 1,4 BD will derivatized with BSTFA as per 5.3.3.

5.2.0 GBL

5.2.1 Extract , or dilute if pure, with chloroform and analyze.

5.3.0 GHB

5.3.1 Extract aqueous samples with chloroform, discard chloroform.

5.3.2 Dry down aqueous layer with nitrogen or dry air. Sample can be warmed to expedite drying as long as the temperature remains below 60 C.

5.3.3 Once sample is completely dry then add 100-200 ul of BSTFA. Cap sample and heat at 60-70C for 15-20 minutes.

5.3.4 Add ethyl acetate and analyze on GC/MS.

6.0.0 FTIR

Aqueous samples are defined as clear, colorless liquids that appear to be water. This doesn't include sodas, sport drinks, etc.

6.1.0 1,4-BD.

If suspected to be pure, run as a liquid sample, i.e. liquid cell, salt windows Gemini, ATR etc.

6.2.0 GBL

6.2.1 If pure, then analyze as a liquid.
6.2.2 If aqueous, extract with chloroform. Discard aqueous layer. Evaporate off chloroform and run as a liquid.

6.3.0 GHB
6.3.1 If solid, analyze as a KBr pellet.
6.3.2 If aqueous, extract with chloroform. Discard chloroform. Evaporate to dryness and run as a KBr pellet.

7.0.0 Scheme

7.1.0 Solids.
7.1.1 Run color test
7.1.2.1 If color test is negative, dissolve in Methanol and analyze on GC/MS.
7.1.2.2 If color test is positive, skip to 7.1.4.
7.1.3 If GC/MS is negative then analysis is complete.
7.1.4 If GC/MS has GBL then derivatize original sample with BSTFA and analyze on GC/MS as per sections 5.3.3 and 5.3.4. Or run sample on FTIR.

7.2.0 Clear, thick liquids.
7.2.1 Place 1-5 mls of the sample in the freezer for fifteen minutes. If it solidifies, extract with methanol and analyze with GC/MS. If results indicate the presence of GBL proceed to section 7.3.4 and 7.3.5.
7.2.2 If sample remains a liquid go to section 7.3.0.

7.3.0 Aqueous samples.
7.3.1 Perform color test.
7.3.2 Acidify a portion of the sample with concentrated H2SO4 and extract with chloroform. Analyze the chloroform layer with GC/MS. If results are negative for GBL then proceed with section 7.3.3. If GBL is present then skip to section 7.3.4. If 1,4 BD is present then report.
7.3.3 If results from 7.3.2 indicate the presence of 1,4 BD then report. If results were negative then take a portion of original sample and dry down with nitrogen and heat (60C). Extract with methanol and analyze with GC/MS.
7.3.4 Take a portion of original sample extract with chloroform. Analyze chloroform layer with GC/MS. Report GBL if found.
7.3.5 Take aqueous layer from 7.3.4 and analyze using sections 5.3.2 through 5.3.4.

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

GAB Rev 1
1/12/07
Replaced
7/3/07

Discipline/Name of Do BD

Revision Number: 1

Issue Date: 01/12/200

APPROVED BY: _____ 1/12/07
Quality Manager Date Signed

#12
GHB, GBL, and 1,4 BD
Analytical Methods

1.0.0 Background

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

The following analytical scheme was developed to separate and identify GHB, GBL, and 1,4 BD while ensuring that GHB is not produced during the process.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of GHB and its related analogs in samples.

3.0.0 Equipment and Reagents

The following pieces of equipment can be used to identify the analytes of interest.

- 3.1.0 A GC/MS and appropriate analytical software. Reference the GC/MS AM.
- 3.2.0 FTIR and appropriate analytical software. Reference the FTIR AM.
- 3.3.0 PH paper
- 3.4.0 ACS grade chloroform, ethyl acetate, methanol, ethanol, H_2SO_4, BSTFA (with 1% TMCS) a TMS derivatizing reagent. *NOTE the BSTFA is available premixed from Cerilliant Corporation.
- 3.5.0 Distilled or deionized water.
- 3.6.0 ACS grade or better of the following solid reagents; bromocresol green, methyl orange, dextrose, aniline hydrochloride, sodium hydroxide. **NOTE, aniline is acutely toxic handle with care.**

4.0.0 Screening Tests

4.1.0 Color Spot Test

A mixture of Bromocresol green, Methyl orange, and Schweppes reagents are tested with samples. A positive reaction for the presence of GHB is one that turns green.

- 4.1.1 Bromocresol green
  Mix 0.03g bromocresol green in 100 mL of 4:1 methanol: water. Adjust to pH 7 with NaOH.
- 4.1.2 Methyl Orange
  Mix 0.01g of methyl orange in 100 mL of methanol. Adjust pH to 7.
- 4.1.3 Modified Schweppes
  Solution A: mix 2.0g dextrose in 20 mL of water.

Solution B: mix 2.4g aniline hydrochloride in 20 mL of ethanol.
Mix solution A & B and dilute to 80 mL with methanol.

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

4.2.0 Physical tests.

4.2.1 Pure GBL and 1,4 BD are viscous liquids at room temperature. 1,4-BD will solidify when placed in a refrigerator (4°C) while GBL will not.

4.2.2 GBL is soluble in chloroform and 1,4 BD is not.

4.3.0 GC/MS

Add concentrated Sulfuric acid to aqueous sample, extract with chloroform and analyze. If GBL is detected then proceed with confirmational GC/MS.

Recipes for other reagents can be found in "*Microgram, Vol XXXV, No.1 January 2002*".

5.0.0 GC/MS Sample Preparation and Analysis

GHB cannot be analyzed directly on a GC/MS as it will convert to GBL in the heated injector port. GHB must be derivatized with BSTFA before injection.

5.1.0 1,4-Butandiol

5.1.1 If pure 1,4 BD is suspected then dilute with methanol and inject into GC/MS.

5.1.2 In aqueous samples, if the concentrations of 1,4 BD are high enough, then the 1,4 BD may be observed in a chloroform extract.

5.1.3 Dry down sample, add methanol, and analyze.

5.1.4 1,4 BD will derivatized with BSTFA as per 5.3.3.

5.2.0 GBL

5.2.1 Extract , or dilute if pure, with chloroform and analyze.

5.3.0 GHB

5.3.1 Extract aqueous samples with chloroform, discard chloroform.

5.3.2 Dry down aqueous layer with nitrogen or dry air. Sample can be warmed to expedite drying as long as the temperature remains below 60 C.

5.3.3 Once sample is completely dry then add 100-200 ul of BSTFA. Cap sample and heat at 60-70C for 15-20 minutes.

5.3.4 Add ethyl acetate and analyze on GC/MS.

6.0.0 FTIR

Aqueous samples are defined as clear, colorless liquids that appear to be water. This doesn't include sodas, sport drinks, etc.

6.1.0 1,4-BD.

If suspected to be pure, run as a liquid sample, i.e. liquid cell, salt windows Gemini, ATR etc.

6.2.0 GBL
- 6.2.1 If pure, then analyze as a liquid.
- 6.2.2 If aqueous, extract with chloroform. Discard aqueous layer. Evaporate off chloroform and run as a liquid.

6.3.0 GHB
- 6.3.1 If solid, analyze as a KBr pellet.
- 6.3.2 If aqueous, extract with chloroform. Discard chloroform. Evaporate to dryness and run as a KBr pellet.

7.0.0 Scheme

7.1.0 Solids.
- 7.1.1 Run color test
- 7.1.2.1 If color test is negative, dissolve in Methanol and analyze on GC/MS.
- 7.1.2.2 If color test is positive, skip to 7.1.4.
- 7.1.3 If GC/MS is negative then analysis is complete.
- 7.1.4 If GC/MS has GBL then derivatize original sample with BSTFA and analyze on GC/MS as per sections 5.3.3 and 5.3.4. Or run sample on FTIR.

7.2.0 Clear, thick liquids.
- 7.2.1 Place 1-5 mls of the sample in the freezer for fifteen minutes. If it solidifies, extract with methanol and analyze with GC/MS. If results indicate the presence of GBL proceed to section 7.3.4 and 7.3.5.
- 7.2.2 If sample remains a liquid go to section 7.3.0.

7.3.0 Aqueous samples.
- 7.3.1 Perform color test.
- 7.3.2 Acidify a portion of the sample with concentrated H_2SO_4 and extract with chloroform. Analyze the chloroform layer with GC/MS. If results are negative for GBL then proceed with section 7.3.3. If GBL is present then skip to section 7.3.4. If 1,4 BD is present then report.
- 7.3.3 If results from 7.3.2 indicate the presence of 1,4 BD then report. If results were negative then take a portion of original sample and dry down with nitrogen and heat (60C). Extract with methanol and analyze with GC/MS.
- 7.3.4 Take a portion of original sample extract with chloroform. Analyze chloroform layer with GC/MS. Report GBL if found.
- 7.3.5 Take aqueous layer from 7.3.4 and analyze using sections 5.3.2 through 5.3.4.

8.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	9/25/03	Original Issue	D.C. Sincerbeaux
1	1/12/07	Added page #'s& history changed sec 3.	D.C. Sincerbeaux

#10
Balance Calibration
Standard Operating Procedures

1.0.0 Background and Scope

In order to ensure the integri[illegible]he reported weights of controlled substances each laboratory within th[illegible] Police system maintains a set of weights that are used to verify t[illegible]ll balances and scales located in each laboratory.

Bal cal Rev 2
5/23/03 - 1/12/07

2.0.0 Equipment

One set of [illegible] weights must be NIST traceable[illegible]

3.0.0 Pr[illegible]

C[illegible].gainst a set of kno[illegible].cure reference. A balanc[illegible], only needs to have its calibratio[illegible] used. Balances not in service do n[illegible]d.

3.1.0 Each ba[illegible].STM weights as reference. This set should [illegible]of samples that will be measured on each balance. [illegible].ypical top loader 1g, 100g, and 2000g weights would b[illegible]e allowable deviation from the standard weights will be 0.0[illegible]%, which ever is greater.

3.2.0 Each laboratory will k[illegible]a log sheet for each balance in use. The log sheet will list the balance identification, the weights used, their indicated weight, whether or not the observed weight is within the tolerance of the balance, the analyst and the date on which the check was performed.

3.3.0 Once a year an independent vendor will calibrate each balance.

3.4.0 An independent vendor will recertify each weight set on a yearly basis.

4.0.0 Consequences

If a balance fails a monthly calibration check, the check is repeated. If the balance still fails then it will be taken out of service until it can be recalibrated or repaired. The balance should be tagged indicating that it is out of service.

Idaho State
Forensic Se

Bal cal Rev 2.1
1/12/07 - 7/3/07

Approval for Quality System

Discipline/Name of Document: # 10 Balance Calibration Verification Analytical Method

Revision Number: 2.1

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager Date Signed

#10
Balance Calibration Verification
Analytical Method

1.0.0 Background and Scope

In order to ensure the integrity of the reported weights of controlled substances each laboratory within the Idaho State Police system maintains a set of weights that are used to verify the calibration of all balances and scales located in each laboratory.

2.0.0 Equipment

One set of ASTM Class 2, or better, weights. These weights must be NIST traceable and certified at the time of purchase.

3.0.0 Procedure

Once a month each balance is to have its calibration checked against a set of certified NIST traceable weights. Results are to be recorded in a log for future reference. A balance that is infrequently used, less than once a month, is required to have a calibration check immediately before the balance is used. Balances not in service are not required to have a calibration check.

3.1.0 Each balance is checked using a set of ASTM weights as reference. This set should span the expected weights of samples that will be measured on each balance. An example; for the typical top loader 1g, 100g, and 2000g weights would be sufficient. The allowable deviation from the standard weights will be 0.01 g or 0.1%, which ever is greater.

3.2.0 Each laboratory will keep a log sheet for each balance in use. The log sheet will list the balance identification, the weights used, their indicated weight, whether or not the observed weight is within the tolerance of the balance, the analyst and the date on which the check was performed.

3.3.0 Once a year an independent vendor will calibrate each balance.

3.4.0 An independent vendor will recertify each weight set on a yearly basis.

4.0.0 Consequences

If a balance fails a monthly calibration check, the check is repeated. If the balance still fails then it will be taken out of service until it can be recalibrated or repaired. The balance shall be tagged indicating that it is out of service.

Revision 2.1
Date issued 1/2007
balcalsop
Issuing Authority: Quality Manager

5.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/26/02	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	5/23/03	Added to section 3.0.0 and 3.1.0	D.C Sincerbeaux
2.1	1/12/07	Added page #s, history & changed wording in 3.0.0 and 4.0.0 "shall", changed title.	D.C. Sincerbeaux

#8
Psilocyn/Psilocybin Mushrooms
Standard Operating Procedures

1.0.0 Background

Psilocyn and psilocybin are related tryptamines that are found in many species of mushrooms. The mushrooms have been used in religious ceremonies for at least 3000 years by the native peoples of Mexico and Central America. Psilocyn and psilocybin are schedule one hallucinogens. More information is ugh the "*Drug Identification Bible 2002*"

Psilocyn Rev 2
9/13/05 - 1/12/07

2.0.0 Scope

The following proc ilocybin from mushrooms.

3.0.0 Equipment and Rea

The following equipr terest.

3.1.0 A GC/MS and C/MS SOP.

3.2.0 Reagent grade

4.0.0 Color Spot Test

4.1.0 "Weber test"

4.1.1 Grind mu

4.1.2 Add grou the addition of a Fast blue BB, or B, turn orange-red within a couple of minutes if psilocin/psilocybin is present.

4.1.3 Remove some of the liquid to another well and then add a drop of concentrated HCl. A positive test is one that turns a blue-green color.

4.1.4 Negative and positive controls need to be run with each batch, and the results documented in the case notes.

5.0.0 GC/MS Sample Preparation and Analysis

5.1.0 Extraction.

5.1.1 Grind up sample with mortar and pestle.

5.1.2 Mix approximately 0.2 grams of sample per 2-3 mls of methanol, cap, shake, and let stand for at least 30 minutes. **NOTE** At this stage the methanolic extract may be injected into the GC/MS.

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

5.1.4 Remove from freezer and immediately add equal volume of acetone and mix.

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

5.2.0 Analysis.

5.2.1 Run samples on GC/MS using a split or splitless data acquisition method depending on the sensitivity of the instrument.

5.2.2 Compare with standard of either psilocyn or psilocybin. NOTE psilocybin breaks down into psilocyn in the hot injection port of a GC.

5.3.0 Conclusions and Reporting.

5.3.1 Confirmation. The retention time must be within 0.04 min of a valid scan of the standard and the MS spectra must match. If both conditions are satisfied then confirmation can be reported as "Contains psilocyn and/or psilocybin".

4.0.0 Thin Layer Chromatography

If differentiation of psilocyn and psilocybin is required then a T1 system (10mls methanol + 7 drops of NH4OH), developed with PDMAB, works well.

6
Lysergic Acid Diethylamide
Standard Operating Procedures

LSD Rev 2
9/2005
replaced
8/17

1.0.0 Background

LSD was originally synthesized from *purpurea.* Street LSD is found most sugar cubes, candies like "Sweet Tar on small pills called microdots. It is presence of light and heat, because o wrapped it metal foil.

2.0.0 Scope

The following analytical procedures acid diethylamide (LSD).

3.0.0 Equipment and Reagents

The following pieces of equipment
- 3.1.0 A GC/MS and appropriate a
- 3.2.0 Ultraviolet light box (short wave UV).
- 3.3.0 Thin Layer Chromatography (TLC) plates and tank.

4.0.0 Ultraviolet (UV) Test

Although by no means definitive, this test can be used as a presumptive test. Place the evidence under the UV. The suspected LSD should glow a light violet-blue. This test is especially useful in identifying which side of a sugar cube, or candy, has been spiked with LSD. It is common for white paper to reflect the UV and appear violet even without LSD.

5.0.0 GC/MS Sample Preparation and Analysis

5.1.0 Sample preparation. As with all GC analyses it may be necessary to concentrate the extracts from either of the following methods; this is done by blowing a stream of air, or other suitable gas, over the top of the solvent. Do not heat!

- 5.1.1 "Window panes", blotter paper, and pulverized microdots can be extracted directly with reagent grade methanol. Place sample in a test tube and add just enough methanol to cover sample. Shake and then let soak for at least an hour. Microdots should soak overnight if possible. Centrifuge if necessary and analyze.
- 5.1.2 Sugar cubes and "Sweet Tarts". Check under UV to find the side that is suspected of being spiked. Scrape off upper layer until approximately one half of the sample, has been used. Dissolve in water and make basic.

Extract with chloroform. Analyze on GC/MS. Using the extraction procedure in 5.2.2, without the derivatizing agent, also works well.

5.1.3 Due to the typically dilute nature of LSD samples, the GC should be set to splitless mode. The injector liner may have to be changed to a splitless model depending on the sensitivity of the particular MS being used. The retention time for LSD is concentration dependent. A series of standards of varying concentrations may have to be run in order to achieve the standard 0.04 minute retention time window.

5.2.0 TMS Derivative

At times, it may be necessary to derivatize weak LSD samples. The following is a summary of one possible method.

5.2.1 Reagents

Ammonium hydroxide (NH4OH)
Methylene chloride, chloroform, or ethyl ether as solvents
MSTFA N-Methyl-N-trimethylsilyl-trifluoroacetamide
BSTFA bis(trimethylsilyl)trifluoroacetamide

5.2.2 Procedure

Place sample in concentrated NH4OH and let soak for at least ten minutes. Add 200 ul of solvent and extract. Separate and evaporate the solvent. Add 30-200 ul of either MSTFA or BSTFA. Analyze on the GC/MS looking for the TMS derivative.

6.0.0 TLC Analysis

A T1 system followed by PDMAB color development works well for LSD. Other appropriate solvent systems, such as chloroform/methanol and acetone, may also be used. After the plate has been spotted with the sample extract, blank, and a standard, and the solvent has risen at least three quarters of the way up, remove the plate and dry. Check with UV and then develop with PDMAB. A purple color should develop with LSD.

6.1.0 The recipe for T1 is 7drops of ammonium hydroxide per 10ml of methanol.

6.2.0 The ratio of chloroform to methanol is 9/1.

6.3.0 PDMAB is 1gram of p'dimethylaminobenzaldehyde in100ml of ethanol and 10ml of conc. hydrochloric acid.

7.0.0 Color Spot Tests

Marquis, grey color
Mandelin's, grey color
PDMAB, purple violet color

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

LSD rev 3
1/12/07
replaced
7/3/07

Discipline/Name of Document: #6 LSD An

Revision Number: 3

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager — Date Signed

6
Lysergic Acid Diethylamide Analytical Method

1.0.0 Background

LSD was originally synthesized from lysergic acid found in the fungus *claviceps purpurea.* Street LSD is found most often on blotter paper. It is also found on sugar cubes, candies like "Sweet Tarts", gelatin squares called windowpanes, and on small pills called microdots. It is most often ingested. It breaks down in the presence of light and heat, because of this the samples are most often found wrapped it metal foil.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of lysergic acid diethylamide (LSD).

3.0.0 Equipment and Reagents

The following pieces of equipment can be used to identify the analyte of interest.

3.1.0 A GC/MS and appropriate analytical software. Reference GC/MS AM.
3.2.0 Ultraviolet light box (short wave UV).
3.3.0 Thin Layer Chromatography (TLC) plates and tank.
3.4.0 All chemicals will be ACS grade or better.

4.0.0 Ultraviolet (UV) Test

Although by no means definitive, this test can be used as a presumptive test. Place the evidence under the UV. The suspected LSD should glow a light violet-blue. This test is especially useful in identifying which side of a sugar cube, or candy, has been spiked with LSD. It is common for white paper to reflect the UV and appear violet even without LSD.

5.0.0 GC/MS Sample Preparation and Analysis

5.1.0 Sample preparation. As with all GC analyses it may be necessary to concentrate the extracts from either of the following methods; this is done by blowing a stream of air, or other suitable gas, over the top of the solvent. Do not heat!

5.1.1 "Window panes", blotter paper, and pulverized microdots can be extracted directly with reagent grade methanol. Place sample in a test tube and add just enough methanol to cover sample. Shake and then let soak for at least an hour. Microdots should soak overnight if possible. Centrifuge if necessary and analyze.

5.1.2 Sugar cubes, "Sweet Tarts" or other candy. Check under UV to find the side that is suspected of being spiked. Scrape off upper layer until approximately one half of the sample, has been used. Dissolve in water

Revision 3
Date Issued 1/2007
Lsdsop
Issuing Authority: Quality Manager

and make basic. Extract with chloroform. Analyze on GC/MS. Using the extraction procedure in 5.2.2, without the derivatizing agent, also works well.

5.1.3 Due to the typically dilute nature of LSD samples, the GC should be set to splitless mode. The injector liner may have to be changed to a splitless model depending on the sensitivity of the particular MS being used. The retention time for LSD is concentration dependent. A series of standards of varying concentrations may have to be run in order to achieve the standard 0.04 minute retention time window.

5.2.0 TMS Derivative

At times, it may be necessary to derivatize weak LSD samples. The following is a summary of one possible method.

5.2.1 Reagents

Ammonium hydroxide (NH_4OH)
Methylene chloride, chloroform, or ethyl ether as solvents
MSTFA N-Methyl-N-trimethylsilyl-trifluoroacetamide
BSTFA bis(trimethylsilyl)trifluoroacetamide

5.2.2 Procedure

Place sample in concentrated NH_4OH and let soak for at least ten minutes. Add 200 ul of solvent and extract. Separate and evaporate the solvent. Add 30-200 ul of either MSTFA or BSTFA. Analyze on the GC/MS looking for the TMS derivative.

6.0.0 TLC Analysis

A T1 system followed by PDMAB color development works well for LSD. Other appropriate solvent systems, such as chloroform/methanol and acetone, may also be used. After the plate has been spotted with the sample extract, blank, and a standard, and the solvent has risen at least three quarters of the way up, remove the plate and dry. Check with UV and then develop with PDMAB. A purple color should develop with LSD.

6.1.0 The recipe for T1 is 7drops of ammonium hydroxide per 10ml of methanol.

6.2.0 The ratio of chloroform to methanol is 9/1.

6.3.0 PDMAB is 1gram of p'dimethylaminobenzaldehyde in100ml of ethanol and 10ml of conc. hydrochloric acid.

7.0.0 Color Spot Tests

Marquis, grey color
Mandelin's, grey color
PDMAB, purple violet color

8.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	11/02/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Scope & add #	D.C. Sincerbeaux
2	9/13/05	6.0.0 added blank	D.C. Sincerbeaux
3	1/12/07	Changed name, added pg #'s & history	D.C. Sincerbeaux

5
Marijuana
Standard Operating Procedures

1.0.0 Background

Marijuana (Cannabis Sativa) has been used for its sedative, euphoriant and hallucinogenic properties for over 3000 years. Written references to it date back to 2700 BC. It is primarily smoked but can be taken orally. The active compound, delta-9-tetrahydrocannabinol (THC) is most concentrated in the resin that is obtained from the flowers of the female plant. It is imperative that the analyst be familiar with the current Idaho code as it pertains to the legal definition of marijuana.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of marijuana in plant material and residue samples. The procedure is composed of a series of tests, none of which by themse[lves] [identify] marijuana or THC, but taken in combination are considered specific[ally]. GC/MS is not routinely applied to marijuana[, but is] specific for THC.

MJ Rec'd 9/13/05 Returned 12/07

3.0.0 Equipment and I[tems]

3.1.0 Stereomic[roscope]
3.2.0 Thin laye[r chromatography]
3.3.0 Aqueous [base] (sodium hydroxide may be used as a substitute)
3.4.0 Petroleu[m ether] (hexane, heptane, and chloroform.
3.5.0 GC/MS

4.0.0 Solvent Extra[ction]

4.1.0 Plant n[aterial]
4.1.1 [ ]ibe.
4.1.2
4.1.3 Use extract [ ]uquenois-Levine.
4.1.4 Retain small amount of unused so[lvent] [as] blank.

4.2.0 Residues
4.2.1. Flush pipe or item(s) containing suspected residue with appropriate solvent and collect solvent in test tube (item(s) may also be swabbed).
4.2.2. Use extract for thin layer and/or modified Duquenois-Levine.
4.2.3. Retain small amount of unused solvent as blank.

5.0.0 Microscopic Examination

5.1.0 Plant material is examined using a stereo microscope for the following characteristics:
5.1.1 Cystoliths and/ or Cystolithic hairs – Small "bear claw" shaped hairs with bases of calcium carbonate. The cystoliths and hairs are located on the

topside of the leaf or leaf- fragment.

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

5.2.0 Seeds are examined using a stereomicroscope for the following characteristics:

5.2.1 Veined shell.

5.2.2 Ridged edges.

5.2.3 Point on one end and dint on the end of plant attachment.

6.0.0 Thin Layer Chromatography

6.1.0 Spot a small amount of solvent extract onto a thin layer plate along side of a marijuana standard and a solvent blank.

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

6.2.1 Hexane/diethyl ether 4:1 (petroleum ether may be substituted for hexane).

6.2.2 Chloroform or Toluene.

6.2.3 Petroleum ether/methanol 95:5 (if PCP is suspected)

6.3.0 Visualize by spraying the plate with Fast Blue BB salt solution.

6.3.0 Compare results of unknown to those of standard. Photocopy the plate for the case file.

7.0.0 Modified Duquenois-Levine

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

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

7.3.0 Add chloroform.

7.4.0 Observe if the purple color transfers into chloroform layer. * Note* Transferring the solution from step 7.2.0 into a clean test tube before the addition of chloroform will decrease the color interference from chlorophyll.

7.5.0 A blank and a standard need to be run with each batch and the results recorded in the case notes.

8.0.0 Results and Reporting

A positive test shall be defined as the following:

8.1.0 Microscopic

8.1.1 Observation of cystolithic hairs on the leaf and/or the presence of characteristic seeds.

8.2.0 Thin Layer

8.2.1 Presence of a red spot with migration distance consistent with the red THC spot of the standard.

8.2.2 Negative blank.

8.3.0 Modified Duquenois-Levine

8.2.1. A purple* color developing after the addition of the HCl (*color may very from blue to reddish purple depending on the sample).

8.2.2. Transfer of the color into the organic layer after the addition of chloroform.

A positive result shall be defined as the following:

8.4.0 Positive microscopic, single TLC system, and modified Duquenois-Levine.
8.4.1 Report as "contains marijuana. Schedule I".

8.5.0 Negative microscopic. Positive modified Duquenois-Levine and two positive TLC systems.
8.5.1 The conclusion should contain the words "contains, marijuana, and resins."

9.0.0 Germination

Marijuana seeds without THC are only controlled if they are fertile. The germination test should only be performed if it has been determined that the seeds do not contain THC. *Note* In determining the presence of THC, soaking the seeds for up to thirty minutes in petroleum ether /hexane, does not effect germination rates.

9.1.0 Wrap a minimum of 10, to a maximum of 100 seeds, in a moist paper towel and place in a covered container. The container is then placed in a safe dark place for 14 days.

9.2.0 Check seeds daily making sure they do not dry out. Also watch out for mold.

9.3.0 Report how many seeds sprouted as a percentage of the original total.

10.0.0 GC/MS Confirmation

10.1.0 Extract sample as in section 4.0.0

10.2.0 Run extract according to GC/MS SOP along with a known standard containing THC.

10.3.0 Compare retention time and ion chromatograph of sample with THC standard.

10.4.0 Report positive results using the words "Contains marijuana. Schedule I" if cystolithic hairs or characteristic seeds were also found, otherwise use 8.5.1.

11.0 References

Identification of Marijuana, by J.I. Thornton and G.R. Nakamura
Journal Forensic Science (1972), 12, 461

History for the Marijuana SOP

Revision #	Issue or review date	History	Author or Reviewer
0	8/17/01	Original Issue	Stuart Jacobson
1	8/27/02	Scope, add #	D.C. Sincerbeaux
2	11/05/04	Small changes in 6.2.3 and 3.4.0, dropped the use of benzene. 8.2.0 red from scarlet. 8.1.0 dropped need for unicellular hairs based on Note inserted into 5.1.2. 6.1.0, 4.1.2, and 4.2.1 appropriate solvent vs. pet ether.	D.C. Sincerbeaux

Approval

Technical Leader _____ *Date:*_____
David Sincerbeaux

QA/QC Manager _____ Date:_____
Rick Groff

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

10 Rev 4
1/12/07
replaced
7/3/07

Discipline/Name of Document: #

Revision Number: 4

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager — Date Signed

5
Marijuana
Analytical Methods

1.0.0 Background

Marijuana (Cannabis Sativa) has been used for its sedative, euphoriant and hallucinogenic properties for over 3000 years. Written references to it date back to 2700 BC. It is primarily smoked but can be taken orally. The active compound, delta-9-tetrahydrocannabinol (THC) is most concentrated in the resin that is obtained from the flowers of the female plant. It is imperative that the analyst be familiar with the current Idaho code as it pertains to the legal definition of marijuana.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of marijuana in plant material and residue samples. The procedure is composed of a series of tests, none of which by themselves are specific for marijuana or THC, but taken in combination are considered specific for the presence of marijuana or its resins. GC/MS is not routinely applied to marijuana analysis but may be used and is considered specific for THC.

3.0.0 Equipment and Reagents

- 3.1.0 Stereomicroscope.
- 3.2.0 Thin layer chromatography tank and plates.
- 3.3.0 Aqueous Fast Blue BB solution. (a Fast Blue B salt solution may be used as a substitute)
- 3.4.0 ACS grade Petroleum ether, hexane, diethyl ether, methanol, toluene, and chloroform.
- 3.5.0 GC/MS and analytical software.

4.0.0 Solvent Extraction

- 4.1.0 Plant material
  - 4.1.1 Place approximately 0.1g of plant material in test tube.
  - 4.1.2 Cover with appropriate solvent.
  - 4.1.3 Use extract for thin layer and/or modified Duquenois-Levine.
  - 4.1.4 Retain small amount of unused solvent as blank.
- 4.2.0 Residues
  - 4.2.1. Flush pipe or item(s) containing suspected residue with appropriate solvent and collect solvent in test tube (item(s) may also be swabbed).
  - 4.2.2. Use extract for thin layer and/or modified Duquenois-Levine.
  - 4.2.3. Retain small amount of unused solvent as blank.

Revision 4
Date Issued 1/2007
Mjsop
Issuing Authority: Quality Manager

5.0.0 Microscopic Examination

5.1.0 Plant material is examined using a stereo microscope for the following characteristics:

5.1.1 Cystoliths and/ or Cystolithic hairs – Small "bear claw" shaped hairs with bases of calcium carbonate. The cystoliths and hairs are located on the topside of the leaf or leaf- fragment.

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

5.2.0 Seeds are examined using a stereomicroscope for the following characteristics:

5.2.1 Veined shell.

5.2.2 Ridged edges.

5.2.3 Point on one end and dint on the end of plant attachment.

6.0.0 Thin Layer Chromatography

6.1.0 Spot a small amount of solvent extract onto a thin layer plate along side of a marijuana standard and a solvent blank.

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

6.2.1 Hexane/diethyl ether 4:1 (petroleum ether may be substituted for hexane).

6.2.2 Chloroform or Toluene.

6.2.3 Petroleum ether/methanol 95:5 (if PCP is suspected)

6.3.0 Visualize by spraying the plate with Fast Blue BB salt solution.

6.3.0 Compare results of unknown to those of standard. Photocopy the plate for the case file.

7.0.0 Modified Duquenois-Levine

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

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

7.3.0 Add chloroform.

7.4.0 Observe if the purple color transfers into chloroform layer. * Note: Transferring the solution from step 7.2.0 into a clean test tube before the addition of chloroform will decrease the color interference from chlorophyll.

7.5.0 A blank and a standard need to be run with each batch and the results recorded in the case notes.

8.0.0 Results and Reporting

A positive test shall be defined as the following:

8.1.0 Microscopic

8.1.1 Observation of cystolithic hairs on the leaf and/or the presence of characteristic seeds.

8.2.0 Thin Layer

8.2.1 Presence of a red spot with migration distance consistent with the red THC spot of the standard.

8.2.2 Negative blank.

8.3.0 Modified Duquenois-Levine

8.2.1. A purple* color developing after the addition of the HCl (*color may very from blue to reddish purple depending on the sample).

8.2.2. Transfer of the color into the organic layer after the addition of chloroform.

A positive result shall be defined as the following:

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

8.4.1 Report as "contains marijuana. Schedule I".

8.5.0 Negative microscopic. Positive modified Duquenois-Levine and two positive TLC systems.

8.5.1 The conclusion should contain the words "contains, marijuana, and resins."

9.0.0 Germination

Marijuana seeds without THC are only controlled if they are fertile. The germination test should only be performed if it has been determined that the seeds do not contain THC. *Note* In determining the presence of THC, soaking the seeds for up to thirty minutes in petroleum ether /hexane, does not effect germination rates.

9.1.0 Wrap a minimum of 10, to a maximum of 100 seeds, in a moist paper towel and place in a covered container. The container is then placed in a safe dark place for 14 days.

9.2.0 Check seeds daily making sure they do not dry out. Also watch out for mold.

9.3.0 Report how many seeds sprouted as a percentage of the original total.

10.0.0 GC/MS Confirmation

10.1.0 Extract sample as in section 4.0.0

10.2.0 Run extract according to GC/MS AM along with a known standard containing THC.

10.3.0 Compare retention time and ion chromatograph of sample with THC standard.

10.4.0 Report positive results using the words "Contains marijuana. Schedule I" if cystolithic hairs or characteristic seeds were also found, otherwise use 8.5.1.

11.0 References

Identification of Marijuana, by J.I. Thornton and G.R. Nakamura
Journal Forensic Science (1972), 12, 461

12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	8/17/01	Original Issue	Stuart Jacobson
1	8/27/02	Scope, add #	D.C. Sincerbeaux
2	11/05/04	Small changes in 6.2.3 and 3.4.0, dropped the use of benzene. 8.2.0 red from scarlet. 8.1.0 dropped need for unicellular hairs based on Note inserted into 5.1.2. 6.1.0, 4.1.2, and 4.2.1 appropriate solvent vs. pet ether. Added 11.0 reference section.	D.C. Sincerbeaux
3	9/13/05	Added 7.5.0	D.C. Sincerbeaux
4	1/12/07	Changed name , added pg #'s	D.C. Sincerbeaux

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

Discipline/Name of Document: #2 GC/MS Analytical Method

Revision Number: 5

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager — Date Signed

2
Gas Chromatograph Mass Spectrometer Analytical Method

1.0.0 Background

The gas chromatograph mass spectrometer (GC/MS) is an analytical instrument that separates and identifies a wide variety of organic compounds based on their mass spectra and retention time data.

2.0.0 Scope

The purpose of this Analytical Method is to layout the basic daily tune, scheduled periodic maintenance, sample preparation, and data interpretation necessary to perform quality analysis using a GC/MS.

3.0.0 Equipment and Reagents

3.1.0 Equipment

- 3.1.1 A GC/MS and corresponding analytical software.
- 3.1.2 Capillary column and data acquisition methods sufficient to separate the analytes of interest.
- 3.1.3 Short and long wave UV light source.

3.2.0 Reagents

- 3.2.1 ACS grade, or better, organic solvents.
- 3.2.2 Standards of the analytes of interest. Standard solutions may be prepared in-house or purchased from a commercial source. They can contain a single analyte or a mixture but all must be authenticated before use in casework.
- 3.2.3 Sodium carbonate and bicarbonate, ACS grade.

4.0.0 Mass Spectrometer Tune

4.1.0 Frequency

- 4.1.1 Using Hewlett-Packard/Agilent software and instrumentation an AUTOTUNE will be run after every major maintenance procedure, i.e. source cleaning or column change. They will also be run whenever a drift from expected values is encountered in the QUICKTUNE, see 4.2.

4.1.2 Using Hewlett-Packard/Agilent software and instrumentation, a successful MS QUICKTUNE, or AUTOTUNE, will be run each day that the instrument is used. A day is defined as a twenty-four (24) hour period starting at the time of the tune. If a sequence of samples will run longer than twenty-four hours then it must be interrupted and a successful QUICKTUNE, and daily standard run before the sequence can continue.

4.2.0 Definition of a Successful Tune (using PFTBA)
Using Chemstation software the following parameters should be met.

4.2.1 Mass assignments within +/- 0.2 AMU of 69, 219, and 502

4.2.2 Peak widths (PW) should be within 0.1 AMU of 0.55.

4.2.3 The relative abundances should show 69 as the base peak, although it might switch with the 219 peak. Under no circumstances should the base peak be anything other than 69 or 219. The relative abundances should be anything greater than 30% for 219, anything higher than 1% for 502.

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

4.2.5 The presence of mass 18 (water) and/or 28 (nitrogen) indicate an air leak into the system. If either mass is above 10% relative abundance then maintenance to repair an air leak is required. The exception to this rule is, one to four hours following the pump down of the system or the refilling of the calibration vial, there may be residual air in the system.

4.3.0 The QUICKTUNE, STANDARD SPECTRA TUNE, and AUTOTUNE printouts shall be initialed by a drug analyst and kept in a logbook.

5.0.0 GC/MS Quality Assurance

5.1.0 For each GC/MS, a standard containing at least one controlled substance will be analyzed on each day that samples are to be run. This standard will be run before any casework is analyzed. If for any reason this standard fails, change of retention time, MS scan etc., then the samples analyzed after the previous standard and before the failed standard are to be considered suspect (for the failed analyte). It will be left to the analyst's discretion whether or not the failure of the standard is germane to each sample and whether the affected samples need to be reanalyzed. The failure of the standard due to instrument failure should be noted in the logbook, along with whatever maintenance that was performed to remedy the situation.

5.2.0 To confirm any substance, there must be a standard of that substance analyzed within twenty-four hours of the sample run.

5.3.0 Sample extracts are not to be concentrated to less than approximately 250ul, the volume of a vial insert.

6.0.0 General Scheduled Maintenance

All non-consumable items that are repaired or replaced must be entered into the maintenance logbook. Entrees into the logbook should include any symptoms of problems along with the status of the system after the repair has been completed.

6.1.0 Daily (consumables). These items are needed to operate the GC/MS system but their replacement, or repair, do not need to entered into the maintenance logbook.
- 6.1.1 Perform Autotune
- 6.1.2 Check and fill solvent rinse vial on autosampler, empty waste solvent vials.

6.2.0 Monthly
- 6.2.1 Run a column efficiency standard (GROB, NP ISO, etc.) and compare to previous month's runs, making sure the same type sample mix is analyzed using the same data acquisition method. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook.

6.3.0 Quarterly, if possible.
- 6.3.1 Check the oil level of the rough pump. Fill if needed and note in logbook. This can only be done if the pump is not running.

6.4.0 Annual.
- 6.4.1 Replace solvent trap, if the part is available, and replace pump oil. Should be done when other maintenance is performed, approximately once a year.

7.0.0 Non-scheduled Maintenance

All non-scheduled maintenance is to be performed on an "as needed" basis as indicated from failure of the autotune, poor chromotagraphy, and or other indications of a system failure. All of these types of repairs will be noted in the maintenance log.

7.1.0 Replace or trim column. After a column has been replaced or trimmed the column efficiency standard will be run.

7.2.0 Clean MSD, replace filaments, gold seal, and injection liner, when needed. Consult with manufacturer's manual or software for cleaning procedure.

7.3.0 Replace electron multiplier if, after repeated cleaning of the source, the mv readings remain at or above 3000.

7.4.0 Replace any part, or system of parts, as necessary.

8.0.0 Data Interpretation

8.1.0 Retention time. A sample's retention time will be considered acceptable if a mass spectral scan of the analyte is within +/- 0.04 min of a matching scan from a known standard. Retention time windows are determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996 ".

8.2.0 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

8.2.1 Identification of the molecular (parent) ion, if normally present. * Note* Some compounds do not have molecular ions in their mass spectra.

8.2.2 Presence of the correct base ion.

8.2.3 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.0.0 Blanks

The purpose of instrument blanks is to check for carry-over between samples, while an extraction blank (negative control) checks the level of contamination of all solvents etc. used in preparing the sample. It is acceptable to use an extraction blank as the instrument blank. For the purposes of this section an internal standard is not considered an analyte of interest.

9.1.0 Frequency. An instrument blank will be run after the daily standard(s) and immediately before each sample(s). An extraction blank is to be prepared and run daily.

9.2.0 Interpretation. A blank run is considered blank if the analyte(s) of interest would not be identified using the above criteria from 8.0.

9.3.0 If a blank has an identifiable analyte of interest then the blank will be rerun or replaced until the analyte of interest cannot be identified. The sample(s) immediately following the suspect blank(s) will be re-extracted and reanalyzed after an acceptable blank has been generated.

10.0.0 Sample Preparation Methods

The following are examples of sample preparation methods for specific substances and or classes of substances.

10.1.0 Cocaine

10.1.1 Samples and standards can be extracted directly using an organic solvent.

10.1.2 Samples and standards can be dissolved in water, or weak acid, and then made basic with Na_2CO_3 or other strong base. Finally the solution is extracted using an immiscible solvent.

10.2.0 Opiates

10.2.1 Heroin

10.2.1.1 Samples and standards are extracted directly into solvent.

10.2.1.2 Samples and standards are dissolved in water, or weak acid, and then made basic with $NaHCO_3$. The solution is extracted using chloroform.

10.2.2 Other Opiates

By far the most prevalent, non-heroin, opiates are found in pills. To analyze these samples see the General unknown AM.

10.3.0 Phenethylamines

Either of two extraction methods can be used depending on the analyst's discretion.

10.3.1 Basic Extraction. Place sample into a test tube. Dissolve with distilled water. Make basic with Na_2CO_3 or other strong base. Extract with petroleum ether, hexane, or other non-water soluble solvent. **NOTE** Amphetamine and methamphetamine basic extracts are volatile. If the extract in the sample vial is allowed to completely evaporate then the analyte may be lost. It is important to recap the sample vial with a new septa if the extract needs to be saved for reanalysis or returned to evidence in a trace case where all of the original sample was used.

10.3.2 Direct extraction. A small amount of the sample is dissolved in methanol or other appropriate solvent. A small amount of Na_2CO_3 may be added to improve chromotagraphy.

11.0.0 Documentation. Only the documentation used to reach the conclusion need be kept in the case-file. These include chromatograms of sample(s), standard(s), library search results, and blank(s).

12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	1/10/03	Add sec 9	D.C. Sincerbeaux
3	9/13/05	Changed 9.0.0, 9.1.0, 9.2.0 and 9.4.0 became 10.0.0	D.C. Sincerbeaux
4	6/30/06	Changed 6.0.0, 6.4.1, dropped 6.2.0, 6.3.2, 6.5.0. Changed 7.0.0 and 7.2.0	D.C. Sincerbeaux
5	1/12/07	Added new sec #10, 12, 5.3	

Added pg #s, changed name,
sec 3, dropped 6.1.3 &4, and minor word changes
throughout D.C. Sincerbeaux

#3
Fourier Transform Infrared Spectrometer Standard Operating Procedure

1.0.0 Background

The Fourier Transform Infrared Spectrometer (FTIR) is an analytical instrument that is used to identify compounds based on their infrared absorption properties. The advantages of using FTIR are that it can differentiate stereo isomers and it is fast. A sample can be analyzed in less than five minutes. The main disadvantage is that in order to produce a high quality result; a sample with a purity of approximately 90% is needed.

2.0.0 Scope

This SOP will describe the routine maintenance and calibration standards necessary to perform quality analysis using a FTIR.

3.0.0 Equipment and Reagents

3.1.0 A FTIR and corresponding analytical
3.2.0 IR grade potassium bromide (KBr). Sh
3.3.0 Hydraulic or other press for making K
3.4.0 Any other sample introduction equipm

FTIR Rev 1
8/02 - 7/1/06

4.0.0 Routine Maintenance

4.1.0 Aside from the normal cleaning of the
sample chamber, a thorough cleaning o
done on an annual basis. Vacuum or spr
instrument making sure not to touch any
4.2.0 Background spectra will be collected be
spectra should be run once every hour w
4.3.0 Monthly calibration check. Using the ma
check of the instrument's performance is done using polystyrene film. This procedure will be performed monthly and after any maintenance. All printouts generated are initialed by the analyst and kept in the maintenance logbook. If the calibration does not pass and /or there is any other symptoms of system failure then consult the manufacture. All maintenance is recorded in a logbook.

5.0.0 Standard Library Preparation

In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard. It is not acceptable to confirm on the basis of a match from a commercially produced library (Georgia State etc.).

5.1.0 Production of valid standard library.

A pure sample of a standard is prepared and analyzed using the same procedures that will be used with an unknown. Once a scan has been produced it can then be stored in an internal library. A match made from this library is acceptable to use for confirmation. Libraries should be made up of all available standards including various salt forms and isomers (d & dl etc.). These standard scans can be produced and entered into the library as they are encountered in casework.

5.2.0 Sample preparation methods are covered under the appropriate analytical method SOP's.

6.0.0 Identification Criteria

If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and the second test, if ran, is positive, then the compound is confirmed.

6.1.0 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

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

FTIR rev 2
7/1/06
replaced
1/12/07

#3
Fourier Transform Infra
Standard Operating

1.0.0 Background

The Fourier Transform Infrared Spectrometer (I [illegible] used to identify compounds based on their infrared absorption properties. The advantages of using FTIR are that it can differentiate stereoisomers and it is fast. A sample can be analyzed in less than five minutes. The main disadvantage is that in order to produce a high quality result; a sample with a purity of approximately 90% is needed.

2.0.0 Scope

This SOP will describe the routine maintenance and calibration standards necessary to perform quality analysis using a FTIR.

3.0.0 Equipment and Reagents

3.1.0 A FTIR and corresponding analytical software.
3.2.0 IR grade potassium bromide (KBr). Should be kept in a desiccator.
3.3.0 Hydraulic or other press for making KBr windows.
3.4.0 Any other sample introduction equipment, i.e. Gemini etc.

4.0.0 Routine Maintenance

4.1.0 Aside from any necessary cleaning of the outside of the instrument, the sample chamber should be cleaned on a monthly basis. At the same time, the desiccant should be checked and replaced if necessary. Both of these checks will be noted in the maintenance logbook.

4.2.0 Background spectra will be collected before any samples are run. Background spectra should be run once every hour when performing batch analysis.

4.3.0 Monthly calibration check. Using the manufacture's procedures, a calibration check of the instrument's performance is done using polystyrene film. This procedure will be performed monthly and after any maintenance. The "System Validation Report" printout is to be initialed by the analyst and kept in the maintenance logbook. If the calibration does not pass and /or there is any other symptoms of system failure then consult the manufacturer. Any maintenance is

recorded in the logbook.

5.0.0 Standard Library Preparation

In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard. It is not acceptable to confirm on the basis of a match from a commercially produced library (Georgia State etc.).

5.1.0 Production of valid standard library.
A pure sample of a standard is prepared and analyzed using the same procedures that will be used with an unknown. Once a scan has been produced it can then be stored in an internal library. A match made from this library is acceptable to use for confirmation. Libraries should be made up of all available standards including various salt forms and isomers (d & dl etc.). These standard scans can be produced and entered into the library as they are encountered in casework.

5.2.0 Sample preparation methods are covered under the appropriate analytical method SOP's.

6.0.0 Identification Criteria

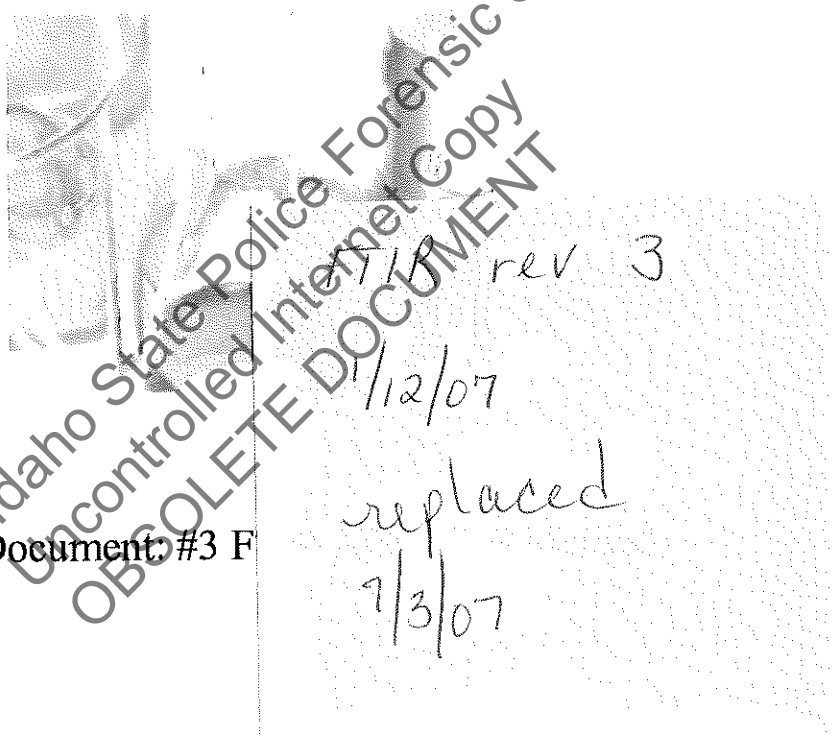
If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and the second test, if ran, is positive, then the compound is confirmed.

6.1.0 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

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

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents



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replaced
7/3/07

Discipline/Name of Document: #3 F

Revision Number: 3

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager — Date Signed

#3
Fourier Transform Infrared Spectrometer Analytical Method

1.0.0 Background

The Fourier Transform Infrared Spectrometer (FTIR) is an analytical instrument that is used to identify compounds based on their infrared absorption properties. The advantages of using FTIR are that it can differentiate stereoisomers and it is fast. A sample can be analyzed in less than five minutes after preparation. The method is limited by the purity of the sample.

2.0.0 Scope

This AM will describe the routine maintenance, performance verification standards, and sample preparation methods necessary to perform quality analysis using a FTIR.

3.0.0 Equipment and Reagents

3.1.0 A FTIR and corresponding analytical software.
3.2.0 IR grade potassium bromide (KBr). Should be kept in a desiccator.
3.3.0 ACS grade solvents.
3.4.0 Hydraulic or other press for making KBr windows.
3.5.0 Any other sample introduction equipment, i.e. Gemini etc.
3.6.0 Reagents
- 3.6.1 Deionized or distilled water
- 3.6.2 $NaSO_4$, ACS grade
- 3.6.3 HCl gas, vapor, 10% solution
- 3.6.4 $NaHCO_3$, ACS grade
- 3.6.5 Na_2CO_3, ACS grade
- 3.6.6 Phenylisothiocyanate, ACS grade

4.0.0 Routine Maintenance

4.1.0 Aside from any necessary cleaning of the outside of the instrument, the sample chamber should be cleaned on a monthly basis. At the same time, the desiccant should be checked and replaced if necessary. Both of these checks will be noted in the maintenance logbook.

4.2.0 Background spectra will be collected before any samples are run. Background spectra should be run once every hour when performing batch analysis.

4.3.0 Monthly performance verification. Using the manufacture's procedures, a performance verification of the instrument is done using polystyrene film. This procedure will be performed monthly and after any maintenance. The "System Validation Report" printout is to be initialed by the analyst and kept in the maintenance logbook. If the verification does not pass and /or there is any other symptom of system failure then consult the manufacturer. Any maintenance is recorded in the logbook.

5.0.0 Standard Library Preparation

In order to confirm the presence of an analyte in a sample, the scan of the sample must match that of a known standard. It is not acceptable to confirm on the basis of a match from a commercially produced library (Georgia State etc.).

5.1.0 Production of valid standard library.

A pure sample of a standard is prepared and analyzed using the same procedures that will be used with an unknown. Once a scan has been produced it can then be stored in an internal library. A match made from this library is acceptable to use for confirmation. Libraries should be made up of all available standards including various salt forms and isomers (d & dl etc.). These standard scans can be produced and entered into the library as they are encountered in casework.

6.0.0 Identification Criteria

If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and the second test, if ran, is positive, then the compound is confirmed.

6.1.0 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

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

7.0.0 Sample Preparation Methods

The following are examples of sample preparation methods for specific substances and or classes of substances.

7.1.0 Cocaine

7.1.1 Pick and Stick. Under a microscope Cocaine HCl appears as flat, mica like crystals. The cocaine can be separated from the cutting agent, added to KBr, and then formed into a pellet. This technique will often yield an IR pure spectra.

7.1.2 Direct. Grind some of the sample with KBr, and form a pellet. This method can be used to determine salt form as long as the sample is relatively pure. Identification

with an appropriate standard is required.

7.1.3 Basic extraction and cleanup. Dissolve sample in water or weak acid. Make basic with $NaHCO_3$. Extract with appropriate non-polar solvent, and dry through $NaSO_4$. Bubble HCl through extract and filter precipitate. Let dry and then mix with KBr, grind, and form a pellet.

7.1.3 Extract with chloroform, or methylene chloride, filter, and then recrystalize.

7.2.0 Heroin

Heroin "Panning" technique from NWAFS newsletter Vol. 12 No. 1. All weights and volumes are approximate.

7.2.1 Dissolve 100mg, preferably more, of sample (Black tar) in 5 mls of 10% HCl. Filter through cotton or glass wool if necessary.

7.2.2 Extract with 5mls of chloroform. Discard the aqueous, acidic, layer.

7.2.3 Back extract with 5 mls of water. Save aqueous layer. Repeat, combining both aqueous layers. Discard chloroform.

7.2.4 Add sodium bicarbonate and extract three times with 5 mls of chloroform. NOTE at this time the solvent can be evaporated onto KBr and analyzed to yield heroin base. May not yield good results due to the polymorphic nature of the base and the base is sticky and it may be difficult to press a good KBr pellet.

7.2.5 Bubble HCl through the chloroform. Dry through a sodium sulfate column. May be analyzed at this stage by drying on KBr.

7.2.6 Using an air stream and heat, recrystalize by doing a solvent exchange with petroleum ether. Evaporate onto KBr.

7.2.7 The spectra of the sample must be compared to a spectra of a standard that was prepared using the same procedure.

7.3.0 Phenethylamines

When purification is necessary the following methods should be used in making KBr pellets. Alternative sample introduction techniques can be used when appropriate.

7.3.1 Amphetamine and Methamphetamine.

Dissolve sample in water or dilute acid. Make basic with Na_2CO_3, or other strong base, and extract with petroleum ether or hexane. Wash extract with water then dry through Na_2SO_4. Bubble HCl gas through solvent and collect the resulting crystals. Wash with additional petroleum ether and let dry. Make KBr pellet. Compare against a known HCl salt standard.

7.3.2 dl-Methamphetamine by PIT derivative.

Dissolve sample in water and make basic with Na_2CO_3. Extract with petroleum ether and dry extract through $NaSO_4$. Add 2 drops of phenylisothiocyanate(PIT) and let stand for 10 minutes. Decant solvent and wash crystals with additional solvent. Dry and make a KBr pellet. Compare with a dl-Methamphetamine standard that was prepared using this procedure.

7.3.3 Direct. Mix and grind sample with KBr. Form into a pellet.

7.3.4 Ephedrine and Pseudoephedrine.

7.3.4.1 Using the same procedure as 7.3.1 will yield the HCl salt.

7.3.4.2 To obtain the base form, dissolve the sample in water, make basic with Na_2CO_3 and extract with petroleum ether or hexane. Evaporate the solvent and make a KBr pellet. Compare against a known base standard.

7.3.4.3 If the sample is a pill(s), crush and add methanol and shake well. Wait approximately one hour and centrifuge. Place supernatant into a clean test tube and allow too evaporate. Take crystals from side of test tube and make a KBr pellet.

8.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add Section 6 and #	D.C. Sincerbeaux
2	6/30/06	Changed Sec 4.1.0	D.C. Sincerbeaux
3	1/12/07	Added 7.0, 8.0, and 3.3 Dropped 5.2, changed title, Backround, reagent list	D.C. Sincerbeaux

2
Gas Chromatograph Mass Spectrometer Standard Operating Procedure

1.0.0 Background

The gas chromatograph mass spectrometer (GC/MS) is an analytical instrument that separates and identifies a wide variety of organic compounds based on their mass spectral and retention time data.

2.0.0 Scope

The purpose of this SOP is to layout the basic daily tune, calibration requirements, scheduled periodic maintenance, and data interpretation necessary to perform quality analysis using a GC/MS.

3.0.0 Equipment, Reagents, and Methods

3.1.0 A GC/MS and corresponding analytical software.

3.2.1 ACS grade, or better, organic solvents

3.2.2 Standards of the analytes of interest. ... ay be prepared in-house or from a com ... alyte or a mixture but all must be au ...

3.2.3 Capillary colu ... separate the analytes of interest.

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7/1/06 - 1/12/07

4.0.0 Mass Spectrometer T ...

4.1.0 Frequency

4.1.1 Using I ... an AUTOTUNE or STANI ... ery major maintenance procedu ... hey will also be run whenev ... ed in the QUICK ...

4.1.2 Using H ... a successful MS QUICK ... that the instrument is used. ... twenty-four (24) hour period starting at the time of the tune. If a sequence of samples will run longer than twenty-four

hours then it must be interrupted and a successful QUICKTUNE run before the sequence can continue.

4.2.0 Definition of a Successful Tune (using PFTBA)
Using HP-Chemstation software the following parameters should be met.

4.2.1 Mass assignments within +/- 0.2 AMU of 69, 219, and 502

4.2.2 Peak widths (PW) should be within 0.1 AMU of 0.55.

4.2.3 The relative abundances should show 69 as the base peak, although it might switch with the 219 peak. Under no circumstances should the base peak be anything other than 69 or 219. The relative abundances should be anything greater than 30% for 219, anything higher than 1% for 502.

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

4.2.5 The presence of mass 18 (water) and/or 28 (nitrogen) indicate an air leak into the system. If either mass is above 10% relative abundance then maintenance to repair an air leak is required. The exception to this rule is, one to four hours following the pump down of the system or the refilling of the calibration vial; there may be residual air in the system.

4.3.0 The QUICKTUNE, STANDARD SPECTRA TUNE, and AUTOTUNE printouts shall be initialed by a drug analyst and kept in a logbook.

5.0.0 GC/MS Quality Assurance

5.1.0 For each GC/MS, a standard containing at least one controlled substance will be analyzed on each day that samples are to be run. This standard will be run before any casework is analyzed. If for any reason this standard fails, change of retention time, MS scan etc., then the samples analyzed after the previous standard and before the failed standard are to be considered suspect (for the failed analyte). It will be left to the analyst's discretion whether or not the failure of the standard is germane to each sample and whether the affected samples need to be reanalyzed. The failure of the standard due to instrument failure should be noted in the logbook, along with whatever maintenance that was performed to remedy the situation.

5.2.0 To confirm any substance, there must be a standard of that substance analyzed within twenty-four hours of the sample run.

6.0.0 General Scheduled Maintenance

Unless specifically exempted, all items that are repaired or replaced must be entered into the maintenance logbook. It is advisable that entrees into the logbook should include any symptoms of problems along with the status of the system after the repair has been completed.

6.1.0 Daily (consumables). These items are needed to operate the GC/MS system but

their replacement, or repair, do not need to entered into the maintenance logbook.

6.1.1 Perform Autotune

6.1.2 Check and fill solvent rinse vial on autosampler, empty waste solvent vials.

6.1.3 Check paper in printer.

6.1.4 Check syringe. Clean or replace if necessary.

6.2.0 Monthly

6.2.1 Run a column efficiency standard (GROB, NP ISO, etc.) and compare to previous months runs, making sure the same type sample mix is analyzed using the same data acquisition method. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook.

6.3.0 Quarterly, if possible.

6.3.1 Check the oil level of the rough pump. Fill if needed and note in logbook. This can only be done if the pump is not running.

6.4.0 Annual.

6.4.1 Replace solvent trap, if the part is available, and replace pump oil. Should be done when other maintenance is performed but not to exceed one year.

7.0.0 Non-scheduled Maintenance

All non-scheduled maintenance is to be performed on an "as needed" basis as indicated from failure of the autotune, poor chromotagraphy, and or other indications of a system failure. All of these types of repairs will be noted in the maintenance log.

7.1.0 Replace or trim column. After a column has been replaced or trimmed the column efficiency standard will be run.

7.2.0 Clean MSD, replace filaments, gold seal, and injection liner, when needed. Consult with manufacturers manual for cleaning procedure.

7.3.0 Replace electron multiplier if, after repeated cleaning of the source, the mv readings remain at or above 3000.

7.4.0 Replace any part, or system of parts, as necessary.

8.0.0 Data Interpretation

8.1.0 Retention time. A sample's retention time will be considered acceptable if a mass spectral scan of the analyte is within +/- 0.04 min of a matching scan from a known standard. Retention time windows are determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996 ". A copy of this method is included in the ISP Controlled Substances SOP manual.

8.2.0 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is

dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

8.2.1 Identification of the molecular (parent) ion, if normally present. * Note* Some compounds do not have molecular ions in their mass spectra.

8.2.2 Presence of the correct base ion.

8.2.3 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.0.0 Blanks

The purpose of instrument blanks is to check for carry-over between samples, while an extraction blank (negative control) checks the level of contamination of all solvents etc. used in preparing the sample. It is acceptable to use an extraction blank as the instrument blank. For the purposes of this section an internal standard is not considered an analyte of interest.

9.1.0 Frequency. An instrument blank will be run after the daily standard(s) and immediately before each sample(s). An extraction blank is to be prepared and run daily.

9.2.0 Interpretation. A blank run is considered blank if the analyte(s) of interest would not be identified using the above criteria from 8.0.

9.3.0 If a blank has an identifiable analyte of interest then the blank will be rerun or replaced until the analyte of interest cannot be identified. The sample(s) immediately following the suspect blank(s) will be reanalyzed after an acceptable blank has been generated.

10.0.0 Documentation. Only the documentation used to reach the conclusion need be kept in the case-file. These include chromatograms of sample(s), standard(s), library search results, and blank(s).

2
Gas Chromatograph Mass Spectrometer Analytical Method

GC-MS Rev 5
1/12/07 - 7/3/07

1.0.0 Background

The gas chromatograph mass spectrometer (GC/MS) is an analytical instrument that separates and identifies a wide variety of organic compounds based on their mass spectra and retention time data.

2.0.0 Scope

The purpose of this Analytical Method is to layout the basic daily tune, scheduled periodic maintenance, sample preparation, and data interpretation necessary to perform quality analysis using a GC/MS.

3.0.0 Equipment and Reagents

3.1.0 Equipment

3.1.1 A GC/MS and correspo

3.1.2 Capillary column and da the analytes of interest.

3.1.3 Short and long wave UV

3.2.0 Reagents

3.2.1 ACS grade, or better, or

3.2.2 Standards of the analytes ared in-house or purchased fr single analyte or a mixtu casework.

3.2.3 Sodium carbonate and bi

4.0.0 Mass Spectrometer Tune

4.1.0 Frequency

4.1.1 Using Hewlett-Packard/Agilent software and instrumentation an AUTOTUNE will be run after every major maintenance procedure, i.e. source cleaning or column change. They will also be run whenever a drift from expected values is encountered in the QUICKTUNE, see 4.2.

4.1.2 Using Hewlett-Packard/Agilent software and instrumentation, a successful MS QUICKTUNE, or AUTOTUNE, will be run each day that the instrument is used. A day is defined as a twenty-four (24) hour period starting at the time of the tune. If a sequence of samples will run longer than twenty-four hours then it must be interrupted and a successful QUICKTUNE, and daily standard run before the sequence can continue.

4.2.0 Definition of a Successful Tune (using PFTBA)
Using Chemstation software the following parameters should be met.

4.2.1 Mass assignments within +/- 0.2 AMU of 69, 219, and 502

4.2.2 Peak widths (PW) should be within 0.1 AMU of 0.55.

4.2.3 The relative abundances should show 69 as the base peak, although it might switch with the 219 peak. Under no circumstances should the base peak be anything other than 69 or 219. The relative abundances should be anything greater than 30% for 219, anything higher than 1% for 502.

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

4.2.5 The presence of mass 18 (water) and/or 28 (nitrogen) indicate an air leak into the system. If either mass is above 10% relative abundance then maintenance to repair an air leak is required. The exception to this rule is, one to four hours following the pump down of the system or the refilling of the calibration vial; there may be residual air in the system.

4.3.0 The QUICKTUNE, STANDARD SPECTRA TUNE, and AUTOTUNE printouts shall be initialed by a drug analyst and kept in a logbook.

5.0.0 GC/MS Quality Assurance

5.1.0 For each GC/MS, a standard containing at least one controlled substance will be analyzed on each day that samples are to be run. This standard will be run before any casework is analyzed. If for any reason this standard fails, change of retention time, MS scan etc., then the samples analyzed after the previous standard and before the failed standard are to be considered suspect (for the failed analyte). It will be left to the analyst's discretion whether or not the failure of the standard is germane to each sample and whether the affected samples need to be reanalyzed. The failure of the standard due to instrument failure should be noted in the logbook, along with whatever maintenance that was performed to remedy the situation.

5.2.0 To confirm any substance, there must be a standard of that substance analyzed within twenty-four hours of the sample run.

5.3.0 Sample extracts are not to be concentrated to less than approximately 250ul, the volume of a vial insert.

6.0.0 General Scheduled Maintenance

All non-consumable items that are repaired or replaced must be entered into the maintenance logbook. Entrees into the logbook should include any symptoms of problems along with the status of the system after the repair has been completed.

6.1.0 Daily (consumables). These items are needed to operate the GC/MS system but their replacement, or repair, do not need to entered into the maintenance logbook.
- 6.1.1 Perform Autotune
- 6.1.2 Check and fill solvent rinse vial on autosampler, empty waste solvent vials.

6.2.0 Monthly

6.2.1 Run a column efficiency standard (GROB, NP ISO, etc.) and compare to previous month's runs, making sure the same type sample mix is analyzed using the same data acquisition method. Retention times should be within +/- 0.04 minutes. A printout is kept in the maintenance logbook.

6.3.0 Quarterly, if possible.
- 6.3.1 Check the oil level of the rough pump. Fill if needed and note in logbook. This can only be done if the pump is not running.

6.4.0 Annual.
- 6.4.1 Replace solvent trap, if the part is available, and replace pump oil. Should be done when other maintenance is performed, approximately once a year.

7.0.0 Non-scheduled Maintenance

All non-scheduled maintenance is to be performed on an "as needed" basis as indicated from failure of the autotune, poor chromotagraphy, and or other indications of a system failure. All of these types of repairs will be noted in the maintenance log.

7.1.0 Replace or trim column. After a column has been replaced or trimmed the column efficiency standard will be run.

7.2.0 Clean MSD, replace filaments, gold seal, and injection liner, when needed. Consult with manufacturer's manual or software for cleaning procedure.

7.3.0 Replace electron multiplier if, after repeated cleaning of the source, the mv readings remain at or above 3000.

7.4.0 Replace any part, or system of parts, as necessary.

8.0.0 Data Interpretation

8.1.0 Retention time. A sample's retention time will be considered acceptable if a mass spectral scan of the analyte is within +/- 0.04 min of a matching scan from a known standard. Retention time windows are determined using the method described in "EPA SW846, method 8000B, section 7.6, Revision 2, December 1996 ".

8.2.0 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

8.2.1 Identification of the molecular (parent) ion, if normally present. * Note* Some compounds do not have molecular ions in their mass spectra.

8.2.2 Presence of the correct base ion.

8.2.3 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.0.0 Blanks

The purpose of instrument blanks is to check for carry-over between samples, while an extraction blank (negative control) checks the level of contamination of all solvents etc. used in preparing the sample. It is acceptable to use an extraction blank as the instrument blank. For the purposes of this section an internal standard is not considered an analyte of interest.

9.1.0 Frequency. An instrument blank will be run after the daily standard(s) and immediately before each sample(s). An extraction blank is to be prepared and run daily.

9.2.0 Interpretation. A blank run is considered blank if the analyte(s) of interest would not be identified using the above criteria from 8.0.

9.3.0 If a blank has an identifiable analyte of interest then the blank will be rerun or replaced until the analyte of interest cannot be identified. The sample(s) immediately following the suspect blank(s) will be re-extracted and reanalyzed after an acceptable blank has been generated.

10.0.0 Sample Preparation Methods

The following are examples of sample preparation methods for specific substances and or classes of substances.

10.1.0 Cocaine

10.1.1 Samples and standards can be extracted directly using an organic solvent.

10.1.2 Samples and standards can be dissolved in water, or weak acid, and then made basic with Na_2CO_3 or other strong base. Finally the solution is extracted using an immiscible solvent.

10.2.0 Opiates

10.2.1 Heroin

10.2.1.1 Samples and standards are extracted directly into solvent.

10.2.1.2 Samples and standards are dissolved in water, or weak acid, and then made basic with $NaHCO_3$. The solution is extracted using chloroform.

10.2.2 Other Opiates

By far the most prevalent, non-heroin, opiates are found in pills. To analyze these samples see the General unknown AM.

10.3.0 Phenethylamines

Either of two extraction methods can be used depending on the analyst's discretion.

10.3.1 Basic Extraction. Place sample into a test tube. Dissolve with distilled water. Make basic with Na_2CO_3 or other strong base. Extract with petroleum ether, hexane, or other non-water soluble solvent. **NOTE** Amphetamine and methamphetamine basic extracts are volatile. If the extract in the sample vial is allowed to completely evaporate then the analyte may be lost. It is important to recap the sample vial with a new septa if the extract needs to be saved for reanalysis or returned to evidence in a trace case where all of the original sample was used.

10.3.2 Direct extraction. A small amount of the sample is dissolved in methanol or other appropriate solvent. A small amount of Na_2CO_3 may be added to improve chromotagraphy.

11.0.0 Documentation. Only the documentation used to reach the conclusion need be kept in the case-file. These include chromatograms of sample(s), standard(s), library search results, and blank(s).

12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	1/10/03	Add sec 9	D.C. Sincerbeaux
3	9/13/05	Changed 9.0.0, 9.1.0, 9.2.0 and 9.4.0 became 10.0.0	D.C. Sincerbeaux
4	6/30/06	Changed 6.0.0, 6.4.1, dropped 6.2.0, 6.3.2, 6.5.0. Changed 7.0.0 and 7.2.0	D.C. Sincerbeaux
5	1/12/07	Added new sec #10, 12, 5.3	

Added pg #s, changed name,
sec 3, dropped 6.1.3 &4, and minor word changes
throughout D.C. Sincerbeaux

#1
General Drug
Standard Operating Procedures

1.0.0 Background / Scope

The following guidelines describe how controlled substance laboratory reports are to be worded, what to do about analytical methods that are no longer, or rarely used, sample and standards destruction, and sampling rules. These guidelines are a natural evolution of rules and procedures that have been used by ISP for years.

2.0.0 Reporting

The choice of words for t ... of the laboratory report should be as brief as poss ... ments.

2.1.1 The container, if ... tc.

2.1.2 Physical descript ... al etc.

2.1.3 Original weight, ... 2.1.6

2.1.4 Conclusion. See ...

2.1.5 Amount used fo ...

2.1.6 Exceptions. Am ... rijuana pipes do not need to be repo ... reported. Volumes of liquids from ... not be reported.

2.2.0 All controlled ... sible. Exceptions are inadequate sar ... s that have recognizable l ... ytical confirmation if a literature search indicates ... stance, Schedule one or two. A sample from each type of two part, unsealed, g... atin type capsules will be analyzed. For the purpose of satisfying the "two test, two sampling" rule, described in 9.2.0, a literature search will be considered a presumptive test.

2.2.1 If a substance is confirmed the report will read "contains XXXXX".

2.2.2 If a substance is present but not confirmed, the report will read "Results of testing are consistent with XXXX, not confirmed".

2.2.3 Non-analytical identifications of pills will read "*source* (PDR, Logo Index, etc.) lists as XXXX".

2.2.4 All controlled substances, Schedules one through four, should be scheduled.

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

2.3.0 Reported sample weights will not exceed the accuracy of the balance used.

2.3.1 In order to alleviate confusion on the part of our customers, conversion between metric and English units of measure should be reported on marijuana cases, when appropriate. Example 90.7g (3.2oz).

2.3.1 "Trace" will be defined as anything less than 0.10 grams.

3.0.0 Sample and Standard Destruction

3.1.0 Sample Destruction. For the purpose of this section a sample will be defined as any case work related extract, solution, or solid that is not returned to evidence. Standards of non-controlled substances will also be treated using these procedures.

3.1.1 Aqueous liquids will be stored in a waste bottle until disposal. Organic solvents will also be stored until disposal.

3.1.2 Disposal of aqueous liquids shall consist of neutralization of pH followed by solidification of remaining liquid with absorbent material (kitty litter etc.). The bottle and solid will then be discarded with normal trash.

3.1.3 Extracted plant material, test tubes, used empty vials, and TLC plates are placed in the disposable glass containers. Once these containers are full, they are stored until the next scheduled drug evidence burn, where they will be destroyed.

3.1.4 Solid (powder) samples can be either washed down the drain or placed in the liquid (aqueous) waste bottle.

3.1.5 Since the amount of a sample used is recorded in the final report (section 2.1.5) no further documentation will be required.

3.2.0 Controlled Substance Standard Destruction. For the purpose of this section, a standard is defined as any controlled substance used as a reference for confirmatory analysis. Standards will be obtained from commercial or governmental sources i.e. (Sigma, Supelco, and DEA).

3.2.1 When a standard needs to be destroyed, i.e. past the expiration date, contamination, or degradation etc., then the standard will be stored until the next scheduled drug burn and destroyed there. Two criminalists will witness the removal of the standards from the laboratory and fill out any necessary paperwork required by the agency conducting the drug burn. The laboratory standard log will indicate when the standard was destroyed. Any DEA forms will also be filled out and turned over to the proper authorities.

3.2.2 If a standard is removed from the laboratory by being totally consumed, accidentally destroyed or spilled, the removal should be witnessed by a second criminalist and both individuals should sign, and date the standard log.

4.0.0 Old Analytical Methods

There are numerous analytical or extraction methods that at one time were used in the Forensic Service laboratory system but because of being replaced by newer technology, or the infrequency of analysis, are no longer performed on a routine basis. These methods do not warrant new (year 2000) written SOP's. If these methods are used, it is to be noted in the case file. Standard QA/QC procedures, including blanks and standards, should be followed when using these methods. The written SOP's, if they exist, of these methods shall be stored at each laboratory. The following list includes some, but by no means all, of the methods that may still have limited use.

d vs. dl-Methamphetamine determination using microcrystaline tests. Modern Microcrystal Tests for Drugs by C.Fulton 1969. Chapter XVII

Mescaline extraction from Peyote. DEA BNDD Manual. Page 78 through 80.

5.0.0 Sampling Rules

Since not all samples are required to be analyzed in a given case, the following guidelines should be used to help the analyst determine which samples will be tested.

5.0.1 A felony charge has priority over a misdemeanor. Example: a gram of cocaine found in a suspect's pocket will be tested while a gram of marijuana found in the same pocket may not be.

5.0.2 A misdemeanor is treated equally to a felony if it is closer to the suspect or was the probable cause for a subsequent search. Example: A gram of marijuana found in a suspect's pocket would be analyzed in addition to a gram of cocaine found in the suspect's car.

5.0.3 If several samples, of different appearance, are submitted as one piece of evidence then each is analyzed to determine the presence of controlled substances. Example: two plastic bags are found on a suspect. One contains a tan powder and the other contains a white powder. Each powder would be tested. Plant materials do not fall under this rule, see 5.0.1 .

5.0.4 The analyst will always strive to provide evidence supporting the highest charge, i.e. trafficking, manufacturing, delivery vs. felony possession vs. misdemeanor possession.

5.1.0 When only a trace level of sample is present, every effort will be made to use less than one half of the sample. If it is necessary to use the entire sample, then any extracts, left over liquids, or residues will be returned to the evidence envelope. It will be estimated on the report how much of the sample was used.

5.2.0 Multiple samples, non-statistical methods.

5.2.1 For less than trafficking amounts. (See appendix) The number of samples necessary to support the charge will be analyzed. Example: If you have five samples and the charge is possession then only one sample needs to be tested. If the charge is intent to deliver then more samples may need to be

tested. Consultation with the prosecutor should determine the number needed. The report will state the total number of samples, the sample weight of the number actually analyzed, the findings, and the amount used.

5.2.2 For trafficking amounts. **ALL** samples will be analyzed until the appropriate trafficking weight is reached. Example: Forty balloons come in, each with about 0.1g of suspected heroin. The analyst will weigh out enough to get to the first trafficking level, 2.0 g, and analyze each.

5.2.3 Pills. After a reference library check, if the pill(s) in a case needs to be confirmed, one pill of each type needs to be analyzed.

5.2.4 For the non-statistical methods then ONLY the results of the samples actually tested can be reported and testified too. No representation as to the content of the other samples is to be inferred.

5.3.0 Multiple samples, statistical method.
If the content of all the samples of a multi sample exhibit, even those samples not actually analyzed, is to be inferred then a hypergeometric sampling scheme will be employed. The ISP Forensic laboratories will use the software from ENFSI for making the calculations as to the number of samples required. This software has been supplied to each laboratory. It is up to each analyst using this method to understand its limitations and the implications.

5.3.1 Count the number of samples.

5.3.2 The ISP system will use 0.9 as the level of "proportion of positives" and 0.95 as the confidence level.

5.3.3 Enter the values from 5.3.1 and 5.3.2 into the excel program.

5.3.4 Analyze the number of random samples from the resulting calculation.

6.0.0 Reagents

For each reagent that is critical to the success of a test, a worksheet recording the following will be maintained; reagents name, recipe, QC method, expected shelf life (if any), date made, name of preparer, manufacturer and lot numbers of ingredients, and results of QC check. All reagents will be checked against known standards and a blank when they are prepared. Reagents that are prepared for one time use, i.e. Weber test, then the lot numbers, QC results etc. are to be documented in the case notes. If the effectiveness of a reagent is verified with each use and the results are documented in the appropriate case files, then no other documentation is required. In order to minimize the waste of expired reagents; those reagents with expiration dates should be made up in quantities that will be consumed before the expiration date.

The following reagents or situations require special attention;

6.1.0 Marquis. This reagent will degrade over time especially when not refrigerated. To ensure reliability, this reagent will be tested once a month with both a positive and negative control. Methamphetamine and ephedrine, or cocaine standards work well as controls. When testing with methamphetamine, the reaction should flash orange immediately. If the orange reaction is slowed the reagent must be replaced.

6.2.0 Secondary amines. Sodium nitroprusside stock solution "A" should be kept in the dark and refrigerated. Shelf life is up to one year.

7.0.0 Authentication of Standards

Before a standard can be used as a reference for casework in must be authenticated. This only has to be done once.

7.1.0 Authentication is performed on the appropriate instrument, either a GC/MS or FTIR.

7.2.0 A standard will be considered authenticated when the Q is greater than 85 %, as compared to a library search. If the Q is less than 85% then two analysts must concur on the validity of the match. Initials of each analyst will be kept on the printout in the standards logbook. Reference libraries can come from any reliable source, i.e. instrument library or scientific journals or publications.

7.3.0 Authentication documentation will be kept for each standard.

8.0.0 Blanks

A reagent (negative control), or solvent (instrument) blank will be run at least once with each batch of analyses. The results will be noted in the case-file. The exception to this is the FTIR background scan, which does not need to be kept. Additional blanks may be run at the analyst's discretion. The results of a reagent blank are considered negative when there is no evidence of contamination from an analyte of interest. Refer to the GC/MS SOP for specific information regarding blanks.

9.0.0 Identification Criteria

9.1.0 General Guidelines. The following identification criteria will be applied to both controlled and noncontrolled substances unless different criteria are listed in separate SOP's.

9.2.0 Testing Rules

9.2.1 For controlled substances two positive tests from two different sampling events will be employed for conformation. One of the tests must provide structural information, i.e. either MS or FTIR. A positive test is defined as one that gives a reaction or result that indicates the presence of the analyte in question. A negative reaction to a color test cannot be used for a positive test even if a negative reaction was expected. Example: a negative reaction of methamphetamine and cobalt thiocyanate even though no color change is expected.

9.2.2 For non-controlled substances i.e. inorganics or cutting agents the second sampling event does not have to be used.

9.3.0 If a sample's MS spectra matches the spectra of a standard, has a retention time within the acceptable time window, and the second test is positive, if ran, then the compound is confirmed.

9.3.1 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

9.3.2 Identification of the molecular (parent) ion, if normally present. * Note* Some compounds do not have molecular ions in their mass spectra.

9.3.3 Presence of the correct base ion.

9.3.4 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.4.0 If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and a second test is positive, then the compound is confirmed.

9.4.1 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

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

10.0.0 Records Retention

The documentation needed to support the conclusion(s) in the report will be kept in the case file. Current batch documentation will be stored in an area of the laboratory known to and accessible to the controlled substances chemists. Examples of batch documentation are GC/MS autotunes.

11.0.0 Abbreviations

Each laboratory will prepare and maintain a list of abbreviations that are used in the case notes. This list will be updated annually and posted in each laboratory.

Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

Rev 8
1/12/07
replaced
7/3/07

Discipline/Name of Document: #1 General Drug

Revision Number: 8

Issue Date: 01/12/2007

APPROVED BY: _____ 1/12/07
Quality Manager Date Signed

#1
General Drug
Analytical Method

1.0.0 Background / Scope

The following guidelines describe how controlled substance laboratory reports are to be worded, what to do about analytical methods that are no longer, or rarely used, sample and standards destruction, and sampling rules. These guidelines are a natural evolution of rules and procedures that have been used by ISP for years.

2.0.0 Reporting

The choice of words for the "Description and Conclusion" section of the laboratory report should be as brief as possible while containing all of the following elements.

2.1.1 The container, if any, i.e. plastic bag, glass vial, paper bindle etc.
2.1.2 Physical description of substance. Powder, liquid, plant material etc.
2.1.3 Original weight, volume, number of pills etc. of sample. See 2.1.6
2.1.4 Conclusion. See 2.2.1 through 2.2.4
2.1.5 Amount used for analysis, or reserved weight. See 2.1.6
2.1.6 Exceptions. Amounts of residue used in the analysis of marijuana pipes do not need to be reported or noted. Weights of liquids are not to be reported. Volumes of liquids and weights of solids from clanlab samples and the amounts used, need not be reported.

2.2.0 All controlled substances analyzed, will be confirmed if possible. Exceptions are inadequate sample size or inability to obtain a standard. Pills that have recognizable logos and/or identification numbers need analytical confirmation if a literature search indicates that they contain a controlled substance, Schedule I or II. A sample from each type of two part, unsealed, gelatin type capsules will be analyzed. For the purpose of satisfying the "two test, two sampling" rule, described in 9.2.0, a literature search will be considered a presumptive test.
2.2.1 If a substance is confirmed the report will read "contains XXXXX".
2.2.2 If a substance is present but not confirmed, the report will read "Results of testing are consistent with XXXX, not confirmed".
2.2.3 Non-analytical identifications of pills will read "*source* (PDR, Logo Index, etc.) lists as XXXX". All therapeutic ingredients will be listed but their relative amounts will not unless it affects the scheduling.
2.2.4 All controlled substances should be scheduled.
2.2.5 Reporting of non-controlled substances shall be left up to the discretion of the analyst.

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2.3.0 Reported sample weights will not exceed the accuracy of the balance used.

2.3.1 In order to alleviate confusion on the part of our customers, conversion between metric and English units of measure should be reported on marijuana cases, when appropriate. Example 90.7g (3.2oz).

2.3.2 "Trace" or "residue" will be defined as anything less than 0.10 grams.

3.0.0 Sample and Standard Destruction

3.1.0 Sample Destruction. For the purpose of this section a sample will be defined as any case work related extract, solution, or solid that is not returned to evidence. Standards of non-controlled substances will also be treated using these procedures.

3.1.1 Aqueous liquids will be stored in a waste bottle until disposal. Organic solvents will also be stored until disposal.

3.1.2 Disposal of aqueous liquids shall consist of neutralization of pH followed by solidification of remaining liquid with absorbent material (kitty litter etc.). The bottle and solid will then be discarded with normal trash.

3.1.3 Extracted plant material, test tubes, used empty vials, and TLC plates are placed in the disposable glass containers. Once these containers are full, they are stored until the next scheduled drug evidence burn, where they will be destroyed.

3.1.4 Solid (powder) samples can be either washed down the drain or placed in the liquid (aqueous) waste bottle.

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There are numerous analytical or extraction methods that at one time were used in the

Forensic Service laboratory system but because of being replaced by newer technology, or the infrequency of analysis, are no longer performed on a routine basis. These methods do not warrant new (year 2000 or latter) written Analytical Methods. If these methods are used, it is to be noted in the case file. Standard QA/QC procedures, including blanks and standards, should be followed when using these methods. The written Analytical Methods, if they exist, shall be stored at each laboratory. The following list includes some, but by no means all, of the methods that may still have limited use.

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5.0.4 The analyst will always strive to provide evidence supporting the highest charge, i.e. trafficking, manufacturing, delivery vs. felony possession vs. misdemeanor possession.

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5.2.1 For less than trafficking amounts. (See appendix) The number of samples necessary to support the charge will be analyzed. Example: If you have five samples and the charge is possession then only one sample needs to be tested. If the charge is intent to deliver then more samples may need to be tested. Consultation with the prosecutor should determine the number needed. The report will state the total number of samples, the sample weight of the number actually analyzed, the findings, and the amount reserved.

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For each reagent that is essential to the success of a test, a worksheet recording the following will be maintained; reagents name, recipe, QC method, expected shelf life (if any), date made, name of preparer, and results of QC check. All reagents will be checked against known standards and a blank when they are prepared. Reagents that are prepared for one time use, i.e. Weber test, the QC results are to be documented in the case notes. If the effectiveness of a reagent is verified with each use and the results are documented in the appropriate case files, then no other documentation is required. In order to minimize the waste of expired reagents; those reagents with expiration dates should be made up in quantities that will be consumed before the expiration date.

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9.2.2 If only one sampling event can be performed on a sample then n-tridecane internal standard is to be added to the extract before analysis on the GC/MS. A blank with internal standard will also be run. For the concentration of the internal standard

refer to the GC/MS Quant AM and scale accordingly.

9.2.3 For non-controlled substances i.e. inorganics, cutting agents and non-scheduled prescription drugs, the second sampling event does not have to be used.

9.3.0 If a sample's MS spectra matches the spectra of a standard, has a retention time within the acceptable time window, and the second test is positive, if ran, then the compound is confirmed.

9.3.1 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

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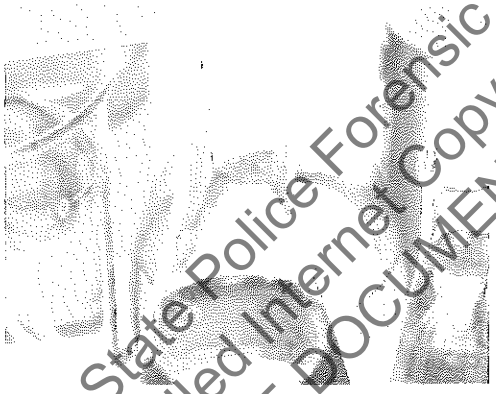
12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1.0	4/26/02	Update section 6	D.C. Sincerbeaux
2.0	7/22/02	Add Sec 7 and 8	D.C. Sincerbeaux
3.0	8/27/02	Add section 9, 10, & #	D.C. Sincerbeaux
4.0	1/10/03	Changed sec 8 and 10	D.C. Sincerbeaux
5.0	4/16/03	Added sec 11.0	D.C. Sincerbeaux
6.0	11/26/03	Changed section 7	D.C. Sincerbeaux
7.0	9/30/05	Major rewrite. Changed sections 1.0.0, 2.1.(2,6), 2.2.0, 3.2.(0,2), 5.2.(1,2,3,), 6.(0,1,2), 8.0.0, 9.2.(0,1,2)	D.C. Sincerbeaux
8.0	12/22/06	Minor word changes throughout, Changed 2.1.6, 2.2.3, 9.2.1, 9.2.2, and 9.2.3	

Rev 9
7/3/07
replaced
7/9/07

Police
ices

Approval trolled Documents



Discipline/Name of Document: Controlled Substances
#1 General Drug Analytical Method

Revision Number: 9

Issue Date: 7/3/2007

APPROVED BY: Carina C Mosley
Quality Manager

7/3/07
Date Signed

#1
General Drug
Analytical Method

1.0.0 Background / Scope

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2.1.3 Original weight, volume, number of pills etc. of sample. See 2.1.6
2.1.4 Conclusion. See 2.2.1 through 2.2.4
2.1.5 Amount used for analysis, or reserved weight. See 2.1.6
2.1.6 Exceptions. Amounts of residue used in the analysis of marijuana pipes do not need to be reported or noted. Weights of liquids are not to be reported. Volumes of liquids and weights of solids from clanlab samples and the amounts used, need not be reported.

2.2.0 All controlled substances analyzed, will be confirmed if possible. Exceptions are inadequate sample size or inability to obtain a standard. Pills that have recognizable logos and/or identification numbers need analytical confirmation if a literature search indicates that they contain a controlled substance, Schedule I or II. A sample from each type of two part, unsealed, gelatin type capsules will be analyzed. For the purpose of satisfying the "two test, two sampling" rule, described in 9.2.0, a literature search will be considered a presumptive test.
2.2.1 If a substance is confirmed the report will read "contains XXXXX".
2.2.2 If a substance is present but not confirmed, the report will read "Results of testing are consistent with XXXX, not confirmed".
2.2.3 Non-analytical identifications of pills will read "*source* (PDR, Logo Index, etc.) lists as XXXX". All therapeutic ingredients will be listed but their relative amounts will not unless it affects the scheduling.

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2.2.4 All controlled substances should be scheduled.
2.2.5 Reporting of non-controlled substances shall be left up to the discretion of the analyst.
2.3.0 Reported sample weights will not exceed the accuracy of the balance used.
2.3.1 In order to alleviate confusion on the part of our customers, conversion between metric and English units of measure should be reported on marijuana cases, when appropriate. Example 90.7g (3.2oz).
2.3.2 "Trace" or "residue" will be defined as anything less than 0.10 grams.

3.0.0 Sample and Standard Destruction

3.1.0 Sample Destruction. For the purpose of this section a sample will be defined as any case work related extract, solution, or solid that is not returned to evidence. Standards of non-controlled substances will also be treated using these procedures.

3.1.1 Aqueous liquids will be stored in a waste bottle until disposal. Organic solvents will also be stored until disposal.

3.1.2 Disposal of aqueous liquids shall consist of neutralization of pH followed by solidification of remaining liquid with absorbent material (kitty litter etc.). The bottle and solid will then be discarded with normal trash.

3.1.3 Extracted plant material, test tubes, used empty vials, and TLC plates are placed in the disposable glass containers. Once these containers are full, they are stored until the next scheduled drug evidence burn, where they will be destroyed.

3.1.4 Solid (powder) samples can be either washed down the drain or placed in the liquid (aqueous) waste bottle.

3.1.5 Since the amount of a sample used is recorded in the final report (section 2.1.5) no further documentation will be required.

3.2.0 Controlled Substance Standard Destruction. For the purpose of this section, a standard (primary, secondary, bench) is defined as any controlled substance used as a reference for confirmatory analysis.

3.2.1 When a standard needs to be destroyed, i.e. past the expiration date, contamination, or degradation etc., then the standard will be stored until the next scheduled drug burn and destroyed there. Two criminalists will witness the removal of the standards from the laboratory and fill out any necessary paperwork required by the agency conducting the drug burn. The laboratory standard log will indicate when the standard was destroyed. Any DEA forms will also be filled out and turned over to the proper authorities.

3.2.2 If a standard is removed from the laboratory by being totally consumed, accidentally destroyed or spilled, the removal should be witnessed by a second criminalist and both individuals should sign and date the standard log.

4.0.0 Old Analytical Methods

There are numerous analytical or extraction methods that at one time were used in the Forensic Service laboratory system. These methods do not have approved Analytical Methods. If an analyst decides that these or other non-approved methods need to be used then the analyst must refer to section 15.4.1.2 of the quality manual for the proper procedures before analysis begins.

5.0.0 Sampling Rules

Since not all samples are required to be analyzed in a given case, the following guidelines should be used to help the analyst determine which samples will be tested.

5.0.1 A felony charge has priority over a misdemeanor. Example: a gram of cocaine found in a suspect's pocket will be tested while a gram of marijuana found in the same pocket may not be.

5.0.2 A misdemeanor is treated equally to a felony if it is closer to the suspect or was the probable cause for a subsequent search. Example: A gram of marijuana found in a suspect's pocket would be analyzed in addition to a gram of cocaine found in the suspect's car.

5.0.3 If several samples, of different appearance, are submitted as one piece of evidence then each is analyzed to determine the presence of controlled substances. Example: two plastic bags are found on a suspect. One contains a tan powder and the other contains a white powder. Each powder would be tested. Plant materials do not fall under this rule, see 5.0.1 .

5.0.4 The analyst will always strive to provide evidence supporting the highest charge, i.e. trafficking, manufacturing, delivery vs. felony possession vs. misdemeanor possession.

5.1.0 When only a trace level of sample is present, every effort will be made to use less than one half of the sample. If it is necessary to use the entire sample, then any extracts, left over liquids, or residues will be returned to the evidence envelope. It will be estimated on the report how much of the sample was reserved.

5.2.0 Multiple samples, non-statistical methods.

5.2.1 For less than trafficking amounts. (See appendix) The number of samples necessary to support the charge will be analyzed. Example: If you have five samples and the charge is possession then only one sample needs to be tested. If the charge is intent to deliver then more samples may need to be tested. Consultation with the prosecutor should determine the number needed. The report will state the total number of samples, the sample weight of the number actually analyzed, the findings, and the amount reserved.

5.2.2 For trafficking amounts. **ALL** samples will be analyzed until the appropriate trafficking weight is reached. Example: Forty balloons come in, each with about 0.1g of suspected heroin. The analyst will weigh out enough to get to the first trafficking level, 2.0 g, and analyze each.

5.2.3 Pills. After a reference library check, if the pill(s) in a case needs to be confirmed, one pill of each type needs to be analyzed.

5.2.4 For the non-statistical methods then ONLY the results of the samples actually tested can be reported and testified to. No representation as to the content of the other samples is to be inferred.

5.3.0 Multiple samples, statistical method.
If the content of all the samples of a multi sample exhibit, even those samples not actually analyzed, is to be inferred then a hypergeometric sampling scheme will be employed. The ISP Forensic laboratories will use the software from ENFSI for making the calculations as to the number of samples required. This software has been supplied to each laboratory. It is up to each analyst using this method to understand its limitations and the implications.

5.3.1 Count the number of samples.

5.3.2 The ISP system will use 0.9 as the level of "proportion of positives" and 0.95 as the confidence level.

5.3.3 Enter the values from 5.3.1 and 5.3.2 into the excel program.

5.3.4 Analyze the number of random samples from the resulting calculation.

6.0.0 Reagents

Unless stated in a separate analytical method, or below, the recipes for reagents found in "*Clarke's Analysis of Drugs and Poisons, 3rd edition*" are to be used.

6.1.0 The following list of color test reagents are approved for use.
Marquis, Cobalt thiocyanate, Liebermann's, Mecke's, Froehde, Fast blue, Duquenois, Simon's (2^{nd} amines), Dille-Koppanyi, and Sulfuric acid/UV.

6.2.0 The following reagents are approved as spray reagents Fast blue, Iodoplatinate, p-DMAB, Fluorescamine, and Dragendorff's.

6.3.0 For each reagent that is essential to the success of a test, a worksheet recording the following will be maintained; reagents name, recipe, QC method, date made, name of preparer, and results of QC check. All reagents will be checked against known standards and a blank when they are prepared. Reagents that are prepared for one time use, i.e. Weber test, the QC results are to be documented in the case notes. If the effectiveness of a reagent is verified with each use and the results are documented in the appropriate case files, then no other documentation is required.

6.4.0 Shelf life. With the exception of Marquis, Cobalt thiocyanate, and Simon's, which

are to be tested monthly, all reagents are to be tested with a positive control and a blank, or negative control as appropriate, with each use. Shelf life is thus considered indefinite.

6.5.0 The following reagents or situations require special attention;

6.5.1 Marquis. This reagent will degrade over time especially when not refrigerated. Test with both a positive (methamphetamine) and negative (dimethyl sulfone) control. When testing with methamphetamine, the reaction should flash orange immediately. If the orange reaction is slowed the reagent must be replaced.

6.5.2 Simon's (2^{nd} amines). Sodium nitroprusside stock solution "1" should be kept in the dark and refrigerated.

6.5.3 A 2% (w/v) cobalt thiocyanate aqueous solution is used for cocaine. Mix cobalt thiocyanate with distilled/deionized water and filter if necessary. Solution should be clear and pink. A positive reaction produces a turquoise blue precipitate. HCl is added to the test well containing the sample and cobalt thiocyanate if the sample is suspected of containing cocaine base. Test with both a positive (cocaine) and negative (dimethyl sulfone) control.

6.5.4 Fast Blue BB salt solution for marijuana and mushrooms. Add enough of the Fast Blue BB salt to distilled/deionized water to change the water to a yellow color. The exact concentration is not relevant as the solution is tested with each use and thus depends on the analyst's personal preference.

7.0.0 Authentication of Standards

Before a standard can be used as a reference for casework, it must be authenticated. This only has to be done once.

7.1.0 Authentication is performed on the appropriate instrument, either a GC/MS or FTIR.

7.2.0 A standard will be considered authenticated when the match (Q) is greater than 85 %, as compared to a library search. If the match is less than 85% then two analysts must concur on the validity of the match. Initials of each analyst will be kept on the printout in the standards logbook or file. Reference libraries can come from any reliable source, i.e. instrument library or scientific journals or publications.

7.3.0 Authentication documentation will be kept for each standard.

7.4.0 Standards will be obtained from commercial or governmental sources i.e. Sigma, Supelco, and DEA, ect. Standards may also be obtained from previously analyzed casework.

8.0.0 Blanks

A reagent (negative control), or solvent (instrument) blank will be run at least once with each batch of analyses. The results will be noted in the case-file. The exception to this is the FTIR background scan, which does not need to be kept. Additional blanks may be run at the analyst's discretion. The results of a reagent blank are considered negative when there is no evidence of contamination from an analyte of interest. Refer to the GC/MS Analytical Method for specific information regarding blanks.

9.0.0 Identification Criteria

9.1.0 General Guidelines. The following identification criteria will be applied to both controlled and noncontrolled substances unless different criteria are listed in separate Analytical Method's.

9.2.0 Testing Rules

9.2.1 For each controlled substance, whenever possible, two positive tests from two different sampling events will be employed for confirmation. One of the tests must provide structural information, i.e. either MS or FTIR. A positive test is defined as one that gives a reaction or result that indicates the presence of the analyte in question. A negative reaction to a color test cannot be used for a positive test even if a negative reaction was expected. Example: a negative reaction of methamphetamine and cobalt thiocyanate even though no color change is expected.

9.2.2 If only one sampling event can be performed on a sample then n-tridecane internal standard is to be added to the extract before analysis on the GC/MS. A blank with internal standard will also be run. Use either a 1000 or 10,000 ug/ml tridecane/ methanol or chloroform stock standard.

9.2.3 For non-controlled substances i.e. inorganics, cutting agents and non-scheduled prescription drugs, the second sampling event does not have to be used.

9.3.0 If a sample's MS spectra matches the spectra of a standard, has a retention time within the acceptable time window, and the second test is positive, if ran, then the compound is confirmed.

9.3.1 Mass spectral interpretation. For the purpose of drug identification, analysis of mass spectra is one of pattern recognition. A great deal of the interpretation is dependent on each analyst's opinion as to what constitutes a match. All comparisons for the purpose of confirmation are made between analytical standards, not library searches, and the sample spectra. The determination of what constitutes a minor peak, and its relative significance, shall be left up to the individual analyst. The following are the minimum requirements to determine a match.

9.3.2 Identification of the molecular (parent) ion, if normally present. * Note Some compounds do not have molecular ions in their mass spectra.

9.3.3 Presence of the correct base ion.

9.3.4 The ratios of the relative abundances of the major ions, from the sample, should be similar to those of the standard.

9.4.0 If a sample's FTIR spectra matches a spectra of a standard that was prepared the same as the sample, and a second test is positive, then the compound is confirmed.

9.4.1 Standard spectra are prepared from authenticated standards and then stored internally for each FTIR instrument, at each laboratory.

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

10.0.0 Records Retention

The documentation needed to support the conclusion(s) in the report will be kept in the case file. Current batch documentation will be stored in an area of the laboratory known to and accessible to the controlled substances chemists. Examples of batch documentation are GC/MS autotunes.

11.0.0 Abbreviations

Each laboratory will prepare and maintain a list of abbreviations that are used in the case notes. This list will be updated annually and posted in each laboratory.

12.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1.0	4/26/02	Update section 6	D.C. Sincerbeaux
2.0	7/22/02	Add Sec 7 and 8	D.C. Sincerbeaux
3.0	8/27/02	Add section 9, 10, & #	D.C. Sincerbeaux
4.0	1/10/03	Changed sec 8 and 10	D.C. Sincerbeaux
5.0	4/16/03	Added sec 11.0	D.C. Sincerbeaux
6.0	11/26/03	Changed section 7	D.C. Sincerbeaux
7.0	9/30/05	Major rewrite. Changed sections1.0.0, 2.1.(2,6), 2.2.0, 3.2.(0,2), 5.2.(1,2,3,), 6.(0,1,2), 8.0.0, 9.2.(0,1,2)	D.C. Sincerbeaux
8.0	12/22/06	Minor word changes throughout, Changed 2.1.6, 2.2.3, 9.2.1, 9.2.2, and 9.2.3	
9.0	7/3/2007	Added 6.3, 6.4,7.4 changed 3.2, 4.0,6.0, 6.1	D.C. Sincerbeaux