Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents

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

Revision Number: 10

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APPROVED BY: Corinna C Owsley
Quality Manager

7/19/07
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. If the charge on a marijuana case is based on the number of plants, then the weight of the sample and the reserve does not need to be recorded. 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.)

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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.

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 (2nd amines), Dille-Koppanyi, and Sulfuric acid/UV.

6.2.0 The following reagents are approved as spray reagents Fast blue, Iodoplatinate, Van Urk (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.
The recipe for Marquis: slowly add 100mls of sulfuric acid to 1ml of approximately 37% (w/w) formaldehyde.

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.

6.5.5 Duquenois. Add 2.5 mls acetaldehyde and 2 g vanillin to 100mls of 95% or greater ethanol.

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 reviewed 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 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	
9.0	7/3/2007	Added 6.3, 6.4,7.4 changed 3.2, 4.0,6.0, 6.1	D.C. Sincerbeaux
10.0	7/19/07	changed 2.1.6, 6.5.1 added 6.5.5	D.C. Sincerbeaux

History for the GC/MS Quantitation SOP

Revision #	Issue or review date	History	Author or Reviewer
0	5/24/02	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	1/10/03	Added 7.7 and 7.8	D.C. Sincerbeaux
3	9/30/05	Added 4.4.0, Changed 5.1.0, 5.2.0, 5.3.0, 4.3.0, 7.3.0 and renumbered 7.0. Added 7.10.0	D.C Sincerbeaux

Approval

Technical Leader _____ Date: 9-30-05
David Sincerbeaux

QA/QC Manager _____ Date: Sept. 29, 2005
Rick Groff

#11
Standard Operating Procedures For the Quantification of Solid Dosage Drugs Using GC/MS with Internal Standards

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. Typically this analysis is performed on casework that will ultimately be tried in federal court.

2.0.0 Scope

Although the following procedures have only been tested using cocaine and methamphetamine, the principals behind them are sound and should allow for the analysis of other controlled substances as long as the appropriate analytical standards, solvents, and internal standards are used.

3.0.0 Equipment and Reagents

3.1.0 Gas Chromatograph/Mass Spectrometer (GC/MS) and corresponding software.
3.2.0 Standards of the analyte of interest.
3.3.0 Appropriate GC or pesticide grade solvent. Chloroform for cocaine and methamphetamine. Use chloroform for mixing with the internal standards as well.
3.4.0 Volumetric flasks (10 and 25ml).
3.5.0 Gas tight syringes in a variety of sizes. (2.5ml, 250ul, and 25ul work well)
3.6.0 Internal standards. n-Tridecane for the phenethylamines and n-Octacosane for cocaine.

4.0.0 Generation of Standard Curve

A linear five-point calibration curve with a correlation coefficient of 0.995, or better, is required. In order to reduce the number of samples that need to be diluted the concentrations of the standards used to generate the curve should span the widest possible range while maintaining linearity.

4.1.0 Accurately prepare a stock solution of your standard at approximately 10,000 ug/ml. Make a solution of the internal standard also at 10,000 ug/ml.
4.2.0 Using the gastight syringes, sample vials, and serial dilutions prepare at least six standards of various concentrations, for example 5000, 2500, 1000, 500, 250, and 100ug/ml. Using at least six levels allows one to be thrown out if it is an outlier instead of remaking another standard.
4.3.0 Place 1.0 ml of each standard in an autosampler vial and add 100ul of the internal standard. **NOTE**: It is recommended that all additions to the vial should be injected through the septa in order to minimize spillage and vaporization of

solvent.

4.4.0 For methamphetamine, the addition of 100 ul of a strong base solution will improve chromatography and is allowed as long as the samples and standards are prepared in exactly the same way.

4.5.0 Using the GC/MS software set up the calibration acquisition parameters and tables. For Hewlett Packard/Agilent Chemstation software the parameters and tables are found in the data analysis/ calibration section.

5.0.0 Sample Preparation

One of the basic requirements in determining an accurate quantification is that the sample must be homogenous. The sample must also be prepared using the same extraction procedure that was used in generating the standard curve.

5.1.0 Initially rough grind the sample with a mortar and pestle until the entire sample will pass through a US No. 4 sieve. Roll and quarter the sample until a subsample* of about 10 grams is obtained. Grind the sub sample until a fine powder is formed. * **NOTE*** If the sample is less than 10 grams then grind the entire sample into a fine powder.

5.2.0 Using an analytical balance that is accurate to at least 0.1 milligram, accurately weigh out a sample of at least 0.1 g and place into a volumetric flask. Add solvent, shake to dissolve, and bring to volume.

5.3.0 Using a 2.5 or 1.25 ml syringe, remove 1.0mls of extract and place in an autosampler vial. Add 100ul of internal standard, and base if used for the curve, and analyze. **NOTE**: It is recommended that all additions to the vial should be injected through the septa in order to minimize spillage and vaporization of the solvent

5.4.0 If a sample's response is greater/lower than the standard response of the highest/lowest point used in generating the curve then the sample must be diluted/concentrated and reanalyzed. **Note:** Ideally the concentration of the sample extract should fall in around the midpoint of the calibration curve.

6.0.0 Calculation of Final Results

Using the equation of the valid curve, calculate the concentration in the vial (the computer software should do this). Use the following equation to calculate the concentration of the analyte in the original sample:

$$\frac{(\text{A ug/ml}) \times (\textbf{Milliliters of solvent} \text{ ..include any dilution factors}) \times 100}{(1000) \times (\textbf{B mg})} = \% \text{ analyte}$$

A = Concentration given by curve
B = Weight of sample used, in milligrams

7.0.0 Notes and QA/QC

7.1.0 The curve must be linear

7.2.0 The area counts of the internal standard should be consistent from the beginning to the end of the run (+/- 10% of the mean).

7.3.0 Because of the rarity of the requests for quantification it would be unusual to need

to run samples more than twenty-four hours after the generation of the curve. If it is necessary to run samples past the twenty fourth hour, a midrange standard will be run and the resultant concentration will be within (+/-) 15 % of the known value. If it is not then the standard can be repeated. A different standard can be substituted and run as well. If it is still outside the 15% range then a new curve needs to be generated.

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

7.5.0 It must be shown that each instrument and each analyst performing the analysis can generate reproducible results.

7.6.0 It is acceptable to use either manual or instrument generated integration. The analyst must be consistent however; the integration method used to generate the curve must be used with the corresponding sample set.

7.7.0 Each instrument that will be used to generate quantitative data will have to have it's own set of control limits.

7.8.0 A positive control will be analyzed each time a curve is generated. The positive control will come from a source other than what was used to generate the curve. If the curve was generated from an in-house stock standard then a commercially prepared standard or another in-house standard from a different lot and prepared by a different analyst is to be used as the positive control. If a commercial standard was used to generate the curve then the positive control can come from either another manufacturer, a different lot, or from an in-house standard.

7.9.0 The accuracy of the curve is validated when the value of the positive control is within +/- 15% of the stated value.

7.10.0 The calibration curve, chromatogram and quantitation report of the daily check standard, chromatogram(s) and quantitation report(s) of all samples, and chromatograms of all applicable blanks are to be kept in the case notes. Chromatograms of standards used to generate the curve do not need to be kept.

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to run samples more than twenty-four hours after the generation of the curve. If it is necessary to run samples past the twenty fourth hour, a midrange standard will be run and the resultant concentration will be within (+/-) 15 % of the known value. If it is not then the standard can be repeated. A different standard can be substituted and run as well. If it is still outside the 15% range then a new curve needs to be generated.

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7.10.0 The calibration curve, chromatogram and quantitation report of the daily check standard, chromatogram(s) and quantitation report(s) of all samples, and chromatograms of all applicable blanks are to be kept in the case notes. Chromatograms of standards used to generate the curve do not need to be kept.

History for the Cocaine SOP

Revision #	Issue or review date	History	Author or Reviewer
0	4/1/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Scope, add #	D.C. Sincerbeaux
2	9/13/05	Dropped 6.2.0 changed 6.3.0	D.C. Sincerbeaux

Approval

Technical Leader _____ Date: 9-12-05
David Sincerbeaux

QA/QC Manager _____ Date: 9-13-2005
Rick Groff

#4
Cocaine
Standard Operating Procedures

1.0.0 Background

Cocaine is one of many related alkaloids that can be extracted from the coca plant (Erythroxylon coca). Cocaine is a DEA controlled substance (CII) and can be identified using several different analytical techniques. General information about cocaine can be found in, "Erythroxylon Coca" a lecture by J.T. Maher 1976, DEA "Cocaine" by C.Van Dyke & R.Byck, Scientific American Vol. 246 number 3, 1982. "Topics in the Chemistry of Cocaine" by H.L. Schlesinger, Bulletin on Narcotics, Vol.XXXVII, No.1, 1985
"Drug Identification Bible", 4th Edition, 1999.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of cocaine in samples.

3.0.0 Equipment and Reagents

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

3.1.0 A GC/MS and appropriate analytical software. Reference GC/MS SOP.
3.2.0 FTIR and appropriate analytical software. Reference FTIR SOP.
3.3.0 Polarizing microscope and reagents. Reference General Drug SOP.

4.0.0 Color Spot Tests

Cobalt thiocyanate is the most common spot test for cocaine. The base form of cocaine will not react with the cobalt thiocyanate. If the base form is suspected then a drop of HCl must be added to the sample. If cocaine is present then the turquoise precipitate will form.

Recipes for this reagent can be found in "Clarke's Isolation and Identification of Drugs"2nd Edition, 1986.

5.0.0 GC/MS Sample Preparation and Analysis

5.1.0 Sample preparation.
- 5.1.1 Samples and standards can be extracted directly using reagent grade solvent.
- 5.1.2 Samples and standards can be dissolved in water, or weak acid, and then made basic with Na2CO3 or other strong base. Finally the solution is extracted using petroleum ether, chloroform, or hexane.

5.2.0 GC/MS analysis. The retention time of the sample should be within 0.04

minutes of a valid MS scan from the daily standard. **NOTE** The GC/MS is sensitive to cocaine and care must be given to not overload the column and detector.

6.0.0 FTIR Sample Preparation Methods

6.1.0 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.

6.2.0 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 appropriate standard is required.

6.3.0 Basic extraction and cleanup. Dissolve sample in water or weak acid. Make basic. Extract with appropriate non-polar solvent, and dry through NaSO4. Bubble HCl through extract and filter precipitate. Let dry and then mix with KBr, grind, and form a pellet.

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