History for the General Drug SOP

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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
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Approval

Technical Leader _____ Date: 11-26-03
David Sincerbeaux

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#1
General Drug
Standard Operating Procedures

1.0.0 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. Not all of these rules will apply to the analysis of marijuana and clandestine laboratory samples. The exceptions for these samples will be noted in their respective SOP's.

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, green plant material etc.
2.1.3 Original weight, volume, number of pills etc. of sample.
2.1.4 Conclusion. See 2.2.1 through 2.2.4
2.1.5 Amount used for analysis, or reserved weight.

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 one or two. All two part, unsealed, gelatin type capsules will be analyzed. For the purpose of satisfying the "two test, two sampling" rule, described in the appropriate analytical SOP's, a literature search will be considered as 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".
2.2.3 All controlled substances should be scheduled if possible.
2.2.4 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 (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 accidentally destroyed in the laboratory, spilled etc. it 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.

5.2.1 For less than trafficking amounts. (See appendix) A number of samples equal to the square root of the total number of samples will be analyzed. Fractional square roots will always be rounded up to the next whole number. Example: If you have five samples, then the square root of five is 2.2, so you would analyze three of the five samples. 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 weighed and screened, up to each appropriate trafficking level. Then a square root of that number of samples will be confirmed analytically. 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, say twenty-one balloons. We will screen all twenty-one, and then analyze a total of five of the samples.

5.2.3 Pills. After a reference library check, if a pill case needs to be confirmed, a composite of the square root of the total number of pills is analyzed. Example: One hundred pills with identical markings are identified in the Logo Index as morphine. Ten of the pills would be ground up, mixed, and the resulting powder analyzed.

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. 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 standards work well as controls. When testing with methamphetamine, the reaction should flash orange immediately before turning brown. If the orange reaction is slowed the reagent must be replaced.

An alternative to a monthly testing scheme is an expiration date of three months. If this alternative is used it is imperative that the reagent be replaced on time. The reagent must be replaced before the expiration date if slowing of the orange flash is noted during casework.

6.2.0 Duquenois. The stock of this reagent will be stored in a refrigerator. This reagent will be tested at least monthly, against a known standard, and a blank.

6.3.0 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, when the standard is first opened.

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 analysis. 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. Refer to the GC/MS SOP for specific information regarding instrument blanks.

9.0.0 Identification Criteria

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

9.2.0 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.

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.

9.3.5 Major spurious ions in a sample must be accounted for. Possible sources of spurious ions can include background, coelluting compounds etc.

9.4.0 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.

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 typically needed to support the conclusion(s) in a 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.

History for the GC/MS Quantitation SOP

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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

Approval

Technical Leader _____ Date: 3-24-03
David Sincerbeaux

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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 will 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 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.
4.1.0 Place 1.0 ml of each standard in an autosampler vial and add 100ul of the internal standard. **NOTE**: all additions to the vial should be injected through the septa in order to minimize vaporization of solvent.

4.3.0 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 Place the entire sample into a mortar and grind with a pestle until a fine powder is formed. Depending on sample and mortal size numerous separate grindings may be required. Any separate grindings will be recombined and then thoroughly mixed.

5.2.0 Take a representative sample and weigh on an analytical balance that is accurate to at least 0.1 milligram, and place into a volumetric flask (0.1 grams into a 10ml flask works well, 0.2g in 25mls is better.). Add solvent, shake to dissolve, and fill to the line.

5.3.0 Using the 2.5 ml syringe, remove 1.0mls of extract and place in an autosampler vial. Add 100ul of internal standard and analyze. **NOTE**: all additions to the vial should be injected through the septa in order to minimize vaporization of solvent.

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

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:

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

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 beginning to the end of the run (+/- 10% of the mean).

7.2.0 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. If it is still outside the 15% range then a new curve needs to be generated.

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

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

7.5.0 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.

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

7.7.0 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 made 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.

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