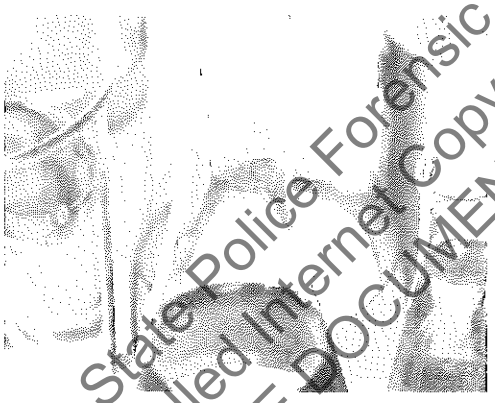
Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents



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

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#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, 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 weights need to be in the notes but do not need to be reported. See 2.1.6

2.1.6 Exceptions. Trace amounts of residue used do not need to be 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 all liquids from clan labs, syringe washes etc., and weights of non-methamphetamine bearing solids from clanlab samples and the amounts used, do not need to be noted. If all of a sample is consumed in analysis and/ or extracts are returned with the evidence, this will be listed on the report.

2.2.0 All controlled substances detected should be confirmed if possible. Exceptions are inadequate sample size, inability to obtain a standard, and compounds that are of the same drug class that have a instrumental response that is relatively minor compared to the major peak and/ or can reasonably be assumed to be a byproduct of the manufacturing process. Examples include, but are not limited to, morphine and codeine in a heroin sample or p2p from a suspected clan lab.

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

2.2.2 If a controlled substance is present but not confirmed, the report will read "Results of testing indicates the presence of a controlled substance, unable to

confirm". The reason why the substance in not confirmed must be in the case record.

2.2.3 Non-analytical identifications of pills will read "*source* (PDR, Logo Index, etc.) lists as XXXX". All therapeutic ingredients will be reported but their relative amounts do not have to be. If the relative amounts effect scheduling then that must be in the notes.

2.2.4 All controlled substances should be scheduled. Exception; liquid samples in unmarked bottles containing a controlled substance, where the schedule of the sample is dependent on the concentration of that controlled substances, should not be scheduled. If a liquid sample comes in a labeled pharmacy bottle and the results of analytical testing confirm the presence of the ingredients on the label, then any schedule on the label should be reported.

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

2.3.0 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 The following conversion factors apply 28.35 g/oz, 453.6 g/lb.

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

2.4.0 The uncertainty of measurement (UM) is: for less than 100g (+/-) 0.01g, for greater than 100g it is 0.03%. See section 12.0 for an explanation of UM

2.5.0 All digits observed from a balance will be reported.

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.

3.2.0 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

Sampling rules allow for the analysis of key evidence items within a case to maximize the resources of the lab. If during the pretrial process it becomes apparent that items not analyzed will require analysis for successful prosecution then upon resubmission that item will receive rush priority.

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 Based on the analysts training and experience if it is suspected different types of felony drugs are submitted then one of each type will be analyzed. The analyst may use resources such as: statements of fact, description of items as well as visual inspection of items in making this determination.

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.

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, and the findings.

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.

5.2.3.1 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. Exception, if a controlled substance has been analytically confirmed from a non-pill sample in the case then a pill(s) listed to contain the same controlled substance only needs a literature search, (section 2.2.3). If a literature search reveals that pills with two, or more, different labels contain the same controlled substance then only one of the pills needs to be analyzed. 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.

5.2.3.2 Acceptable literature references are, published books (PDR, DIB, Logo index etc.) and manufacturers web sites. All literature searches shall be documented with a hard copy (photo copy, printed computer page etc.). Information from Poison control centers can be used as a preliminary test when further analytical testing is performed.

5.2.3.3 If an analyst, through training and experience, can make an educated assumption as to an identity of a partial pills content, that is subsequently analytically confirmed, then the results of a literature search of the partial pill can be used as a presumptive test. Examples of these type of pills are four part bar shaped Xanax, large four part Methadose 40, round Valium heart shaped center holes etc. For the purpose of this section only, if the contents of the partial pill yield a positive color test then the results of the color test should be used instead of the literature search.

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. Exception, for cocaine the base ion of the sample does not have to match that of the standard but does have to be present in significant abundance.

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.

12.0.0 Uncertainty of Measurement

Of the many possible variables that contribute to the uncertainty of measurement, using our AM's, only one is accurately measurable, the use of a balance. If requested by our clients, the contribution of that variable is what we will report. All analysts must be aware of other possible variables and to be able to explain their potential impact on the reported weight. Examples of other variables include but are not limited to, moisture/solvent content, static, and the ability to remove all of a sample from packaging.

13.0.0 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
10.0	7/19/07	changed 2.1.6, 6.5.1 added 6.5.5	D.C. Sincerbeaux
11.0	7/29/08	added 12, 2.4-6, 5.2.3.1 &2. Edited several sections dropped 3.1.5	D.C. Sincerbeaux
12.0	6/22/2010	changed 2.1.6, 2.20, 2.2.2, 2.2.4 added 5.2.3.3	D.C.Sincerbeaux