#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, sampling rules, recipes for color reagents, identification criteria and the FTIR. These guidelines are a natural evolution of rules and procedures that have been used by ISP for years.

2.0.0 Reporting

The laboratory notes shall contain the following:

2.1.1 A description of the proximal container, if any, i.e. plastic bag, glass vial, paper bindle etc.

2.1.2 Physical description of evidence. Powder, liquid, plant material, etc. Including color, shape, and any imprint or writing on pills/tablets/capsules. Documenting score lines is optional. Documenting the description can be done by words, photos or drawings.

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 shall not be reported... Weights of samples from clan labs do not need to be reported but should be noted if the sample appears to contain a significant amount of methamphetamine. If more than half of the visible residue of a sample is to be consumed in analysis then any extracts must be returned with the evidence. Any extracts or washes from evidence that did not have visible residue will also be returned to evidence. This will be listed on the report. If the nature of the sample precludes an accurate weighing (sticky, sludge, moldy etc.) then the sample does not need to be weighed but the reason why must be reported.

2.1.7 All schedule 1 narcotic and schedule 2 controlled substances 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 an 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 In the case of a sample that has a mixture of compounds from different schedules it is up to the analyst to determine if a schedule 1 non-narcotic and/or schedule 3-5 compound warrants confirmation. If it does not then the report should note that the sample indicates the presence of another controlled substance that was not confirmed.

2.2.2 If a controlled substance is present but not confirmed, the report will read "indicates the presence of a controlled substance, not confirmed". The reason why the substance was not confirmed must be on the report.

2.2.3 Non-analytical identifications of pills will read "*source, including year* (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 substance(s) 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 assist our customers with the conversion between metric and English units of measure on marijuana cases, the following statement can be added to the report: 3oz = 85.05g, 1lb = 453.6g

2.3.1 "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 (+/-) it is 0.05g, for greater than 100g it is 0.13g. See section 13.0 for an explanation of UM

2.4.1 When the total weight of a sample(s) falls within the window of uncertainty at regulatory limits, then the uncertainty associated with each weighing event must be listed on the report. Current windows:

For Cocaine and Methamphetamine:
27.50 to 28.50g
198.70 to 201.30g
398.70 to 401.30g

For Heroin:
1.50 to 2.50g
6.50 to 7.50g
27.50 to 28.50g

For Marijuana:
84.65 to 85.45g
452.29 to 454.89g (1 pound)
2266.65 to 2269.25g (5 pounds)
11338.45 to 11341.05g (25 pounds)

2.4.2 The measurement result shall include the measured quantity value (X) along with the associated expanded uncertainty (U), and this measurement shall be reported as X (+/-) U where U is consistent with the units of X, i.e. 28.05g (+/-) 0.04g. On the report (additional notes section) we have to reference that the uncertainty was calculated at the 95% confidence level.

2.4.3 The total U is a product of the calculated uncertainty and the number of weighing events. For example:

0.2=0.04(5) for less than 100grams with five weighings.

2.5.0 All digits observed from a balance will be reported or out to the hundredths place, whichever is less.

2.6.0 In order to report "No controlled Substances detected", at a minimum, a sample must be run on the GC/MS using a temperature program and extraction scheme that encompasses a wide range of drugs. Refer to the general unknown AM.

2.7.0 In multi item samples the report must clearly state what and how many items were tested, i.e. "Three M365 pills, analyzed one….."

2.8.0 If an analytical scheme is employed that is restricted to a limited range of compounds then that limitation must be clearly stated on the report along with any qualifiers. Example: "no basic drugs detected" qualifier."examples of basic drugs include opiates, amphetamines and cocaine."

2.8.1 If a wash sample tests positive for drug XXX, and nothing is detected in the control, then for the control report "No XXX was detected", if the control was run on a restricted scheme as well.

2.9.0 If any drug is detected in the control sample, it must be reported.

2.10.0 For synthetic cannabinoids that have ambiguous scheduling report out " XXXX, a synthetic cannabinoid".

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. Requests for analysis on items not originally tested will be reviewed and prioritized on a case by case basis.

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. 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.), manufacturer's web sites, Drugs.com, and labels from pharmaceutical packaging (ampoules, vials, blister packs, etc.) . All literature searches shall be documented with a hard copy (photo copy, printed computer page etc.). Information from Poison control centers and non-manufacturers' web sites can be used as a preliminary test when further analytical testing is performed or in conjunction with published books or approved websites to delineate pills with similar imprints and descriptions.

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, these values must be listed on the report. A copy of the calculation table from ENSFI will be kept in the case notes.

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 (2nd 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 including but not limited to instrument libraries, scientific journals, or publications. When comparison to a journal, compendium or other document is not an option, mass spectral interpretation may be used in conjunction with the COA (certificate of analysis). This would apply in cases where instrumental data for a drug metabolite is not yet published, but a structurally similar compound is available to assist with interpretation. A second trained analyst must also review and initial the printout verifying the interpretation.

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, etc. 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. Refer to the GC/MS Analytical Method sections 8.0.0 and 9.2.0 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 non-controlled 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. 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, some compounds have several ions that depending on spectral shifting may change base ions, cocaine is an example of this. In these cases 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.

10.0.0 FTIR

10.1.0 The Fourier Transform Infrared Spectrometer (FTIR) is an analytical instrument that is used to identify compounds based on their infrared absorption properties.

10.2.0 Equipment and Reagents

10.2.1 A FTIR and corresponding analytical software.

10.2.2 IR grade potassium bromide (KBr). Should be kept in a desiccator.

10.2.3 ACS grade solvents.

10.2.4 Hydraulic or other press for making KBr windows.

10.2.5 Any other sample introduction equipment, i.e. ATR, Gemini etc.

10.3.0 Routine Maintenance

10.3.1 The desiccant should be checked monthly. If the desiccant is replaced it will be noted in the maintenance log.

10.3.2 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" or "Valpro Qualification 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, perform a bench alignment and or consult the manufacturer. Any maintenance is recorded in the logbook.

10.4.0 Background spectra will be collected immediately before every sample.

10.5.0 Standard Library Preparation

10.5.1 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.).

10.5.2 Production of valid standard library.
A pure sample of a standard is analyzed using the same procedures that will be used with an unknown (ATR vs. KBR etc.). A separate library for each type of sample introduction technique will need to be produced. 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. These standard scans can be produced and entered into the library as they are encountered in casework.

10.6.0 Identification Criteria

10.6.1 If a sample's FTIR spectra matches a spectra of a standard and the second test, if ran, is positive, then the compound is confirmed.

10.6.2 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.

10.6.3 If spectral subtraction is done then what was subtracted needs to be in case notes.

10.6.4 If a FTIR spectra is inconclusive or negative for a controlled substance then the sample will be analyzed on a GC/MS

11.0.0 Records Retention

11.1.0 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.

11.2.0 GC/MS and FTIR data files will be backed up monthly.

12.0.0 Abbreviations

Each laboratory will prepare and maintain a list of abbreviations that are used in the case

notes.

13.0.0 Uncertainty of Measurement on Qualitative Samples

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. It must be clear that that uncertainty number applies only to the act of weighing and not to the sample as a whole. 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. A set of sample pillows has been provided to each lab. These need to be weighed and recorded on a monthly basis. Once a year the UM for the balances will be recalculated and any change will be published.

14.0.0 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, 2.2.0, 2.2.2, 2.2.4 added 5.2.3.3	D.C.Sincerbeaux
13	9/9/2011	Change to 9.2.2, 2.1.6, 2.2.0, 2.3.0, 5.2.1, 5.2.3.2, 5.3.2, 8.0.0 Added 2.2.0.2 and 2.2.0.3	D.C. Sincerbeaux
14	7/2/2012	Changed 1.0.0, 2.2.2, 5.3.2, 13.0.0 and reordered numbers after 9.0. Added 2.6.0, 2.7.0, 2.8.0, 11.1.2 and new section 10.0	D.C. Sincerbeaux
15	2/25/13	Changed 2.0.0, 2.1.1, 2.1.2, 2.1.6, 2.8.0, 5.2.3.2, 9.3.3, Added 2.9.0, 2.10.0	D.C. Sincerbeaux
16	12/20/13	Changed (reporting in) 2, 2.4.0, 5.2.3.2, 13.0.0 Added 2.4.1, 2.4.2, 2.4.3	D.C. Sincerbeaux
17	12/1/14	changed 9.2.2, added 2.10.0	D.C. Sincerbeaux
18	12/16/14	changed 2.1.6, updated 2.4, changed numbering in 2.	D.C. Sincerbeaux