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DRUG CHEMISTRY

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GENERAL INTRODUCTION

The Chemistry Procedures Manual contains common methods and techniques that can be used in the analysis of Controlled Substances and Cannabis. Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

The analysis of controlled substances depends on the type of sample material submitted for analysis. This can be a powder, liquid, licit or illicit tablets or capsules, plant material or residue. The choice of methods used for the analysis of this wide variety of different materials will vary with each case. Because of this, a specific procedure devoted to each drug is neither reasonable nor possible. Rather, the methods used in an analysis will be specified in a general manner for use by the examiner in the analysis of a particular sample.

Sample preparation consists of both mass determination and sampling techniques. Consideration should be given to each before conducting analytical tests. See Appendix II, Appendix III, Appendix IV (sections A-E), and Appendix V for policies regarding both of these areas.

After the mass is determined, the analyst must decide which method will be chosen to perform a preliminary or screening test. This includes:

1. Physical identification by a reference source for legitimate preparations
2. Color Test
3. Thin-Layer Chromatography (TLC)
4. Gas Chromatography (GC)

Less commonly, screening can be done by Infrared Spectroscopy or Gas Chromatography/ Mass Spectrometry or other acceptable analytical tests.

After the preliminary test is performed, a second test, such as Infrared Spectroscopy (IR) and/or Mass Spectrometry (MS) which gives structural information sufficient for the positive identification of the compound, will be utilized. The use of one preliminary and one structural test for the identification of a controlled substance is a minimum. If more tests are required to positively identify the material, an examiner is required to perform them to reach a conclusion as to whether a controlled substance is present or not.

For identification criteria and other guidelines for drug analysis see Appendix IV sections A through E.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: PHYSICAL IDENTIFICATION

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INTRODUCTION

The Physical Identification Procedure can only be used on pharmaceutical preparations. These preparations must be from a commercial manufacturer. Tablets and capsules of clandestine manufacture or counterfeit cannot be identified with this procedure. A positive identification obtained by comparing to a listed or similar reference can be used as a preliminary test on a standard commercial dosage unit.

SAFETY CONSIDERATIONS

Standard Laboratory Practices.

PREPARATIONS

None Required.

INSTRUMENTATION

Generally none required, however, a stereoscope may be useful in identifying numbers or symbols inscribed on the products.

MINIMUM STANDARDS AND CONTROLS

The reference used and the result will be documented in the analytical notes packet. The reference should include the year of the reference (e.g. PDR) or the number/volume and page number (e.g. Identidex). For webpages, a copy of the source (e.g. PDF) must be uploaded into LIMS. The date the webpage was accessed, URL, etc. must also be documented in the analytical notes packet.

When Physical Identification is used as a reference to determine the potential contents of a tablet, it is permissible to use the conclusion "Not Analyzed" provided no further analysis is performed. A conclusion of "Physically resembles a pharmaceutical preparation; no further analysis" is also allowed, but not required. For instances where the tablets can be seen through the evidence packaging, Physical Identification can be used as a reference in the analyst's notes without being included on the report.

PROCEDURE OR ANALYSIS

I. VISUAL EXAMINATION

The chemist should make a thorough visual examination of the material submitted for analysis. This may include the use of the stereoscope. Notes should include a description of the following characteristics, if applicable

Color: A description of the color.

Shape: A description which may include a drawing.

Markings: A description which may include a drawing of the markings found on the tablet or capsule.

Other: Additional observations may include: condition of the material, edge shape and size of the tablet or capsule, and any other relevant observations.

II. LITERATURE COMPARISONS

The use of identification guides is suggested in the case of pharmaceutical preparations. The unknown is compared to a known pharmaceutical or a representation description/picture of a known pharmaceutical. The information gained can be considered in much the same way as the information gained by any examination of external characteristics. The results provide preliminary or presumptive information.

When doing a physical identification on a dosage form, care must be taken to ascertain that the drug has not been tampered with and that it is of legitimate, as opposed to clandestine, origin. The markings, shape, size, color, etc. must be the same as shown/described in the reference.

Optical activity may be determined from a legitimate reference source or identification guide (e.g. PDR). If the reference/guide notates optical rotation, then no further testing for optical rotation need be performed. Propoxyphene is an example of this type of identification. However, the report must specify dextropropoxyphene as required from the Controlled Substance Act.

1. The Physicians' Desk Reference (PDR)
2. Identidex
3. The Drug Identification Bible
4. Redbook
5. The Logo Index
6. RX-ID
7. Drugs.com

Other published materials similar to the above listed references may also be appropriate.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. The Physicians' Desk Reference; Medical Economics: Data Productions, Montvale, NJ,

2. Identidex® System; Micromedex:, Thomson Reuters, 6200 S. Syracuse Way, Suite 300, Englewood, CO.

3. The Drug Identification Bible: Amera-Chem: P.O. Box 518, Grand Junction, Colorado.

4. Redbook; Thomson PDR: Montvale, NJ.

5. The Logo Index for Tablets and Capsules; Drug Enforcement Administration, Office of Forensic Sciences: Washington, D.C.

6. RX-ID; Amera-Chem: P.O. Box 518, Grand Junction, Colorado.

7. Drugs.com, National Library of Medicine, Truven Health Analytics and Cerner Multum, Inc. All Rights Reserved. Copyright 2017

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DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: COLOR AND FUNCTIONAL GROUP

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INTRODUCTION

Color or spot tests are non-specific preliminary screening tests that respond to particular functional groups or a molecular moiety. They are not a positive identification test. They are, however, a good screening or presumptive test because they indicate the type of compound present and give clues as to its structure.

There is always a certain amount of subjectivity that must be taken into account when a color is reported. It is not uncommon for two chemists to describe the same color differently. Aside from the differences in reporting colors that can be attributed to the analyst, colors can also be influenced by the concentration of the solvent or solute or by the presence of diluents or contaminants. Also, the length of time during which the colors are observed may influence the color reported because color transitions and instabilities are not unusual. Allowances should be made for these differences, especially with street samples, where neither the concentration of the drug nor the presence or composition of any diluent or contaminant is known.

SAFETY CONSIDERATIONS

Precaution should be taken when preparing or handling color test reagents. Several reagents contain strong acids which can penetrate clothing or lab coats and cause injuries if they come in contact with skin. These reagents can also splatter, effervesce, or emit noxious or harmful vapors. For these reasons, the reagents should be prepared and the tests should be conducted in the hood while wearing proper Personal Protective Equipment (PPE).

PREPARATIONS

Some common reagent formulations are given on the following pages. The outline also includes information for the type of drug that reacts with each specific reagent. The intended uses are not an exhaustive list. Where possible, American Chemical Society (ACS) grade of chemicals should be employed when preparing in-house reagents. The amounts of reagent used in testing the samples are suggested guidelines and can vary.

CHEN'S TEST

Intended Use:	Non-controlled stimulants and ephedrine
Formulation:	A. 1% acetic acid B. 1% copper sulfate in water C. 2N sodium hydroxide Add 1 drop of each, in order, to sample.
Positive:	Purple
Stability:	Stable
Notes:	Run blank since reagent mixture is light blue.

COBALT THIOCYANATE

Intended Use: Cocaine
Formulation: 2 g cobalt thiocyanate
100 mL water
Positive: Blue
Stability: Very stable
Notes: The free base may cause false negatives. Adding a drop of concentrated HCl to give the HCl salt will often prevent this problem. There is a large group of false positives.

COBALT THIOCYANATE-ACIDIFIED

Intended Use: Cocaine
Formulation: 20 g cobalt thiocyanate
1 L distilled water
40 mL concentrated HCl
Positive: Blue
Stability: Stable
Notes: There are few false positives. The salt form may produce a weak blue.

DILLE-KOPPANYI

Intended Use: Barbituric Acid derivatives
Formulation: A. 0.1 g cobalt acetate
100 mL methanol
0.2 mL glacial acetic acid

B. 5 mL isopropylamine
95 mL methanol

Use 2 drops A and 1 drop B.
Positive: Purple
Stability: Very stable when stored as two solutions.
Notes: There are very few false positives. The sodium salt of the Barbituric Acid will give a violet color at step one.

EHRLICH'S

Intended Use: LSD and hallucinogens
Formulation: 5 g paradimethylaminobenzaldehyde
100 mL concentrated HCl

If necessary, 95% ethanol can be used to dilute this formula to prevent charring.

For spray, dilute Ehrlich's reagent 1:1 with ethanol.
Positive: Purple
Stability: Stable
Notes: There are very few false positives.

FROEHDE'S

Intended Use: Phenols, especially narcotics
Formulation: 50 mg molybdic acid or sodium molybdate
10 mL hot concentrated sulfuric acid

The solution should be colorless.
Positive: Red-purple
Stability: Stable

LIEBERMANN'S

Intended Use: Amphetamines
Formulation: 10 g potassium nitrite or sodium nitrite
100 mL concentrated sulfuric acid

Note: Keep reagents cold when preparing.
Positive: Yellow, gold, bright colors
Stability: Stable

MANDELIN'S

Intended Use: Narcotics, amphetamines, hallucinogens
Formulation: 1 g ammonium vanadate
100 mL concentrated sulfuric acid
Positive: Green, purple, red-purple
Stability: Stable

MARQUIS (LeRosen)

Intended Use:	Amphetamines, narcotics, hallucinogens
Formulation:	8-10 drops 40% formaldehyde 10 mL concentrated sulfuric acid
Positive:	Orange, purple, bright colors
Stability:	Volatile due to the formaldehyde. Very sensitive to water, store in dry place. Limited shelf life.

MECKE (Lafon)

Intended Use:	Alkaloids
Formulation:	0.25 g selenious acid 25 mL concentrated sulfuric acid
Positive:	Green, blue
Stability:	Stable

SANCHEZ

Intended Use:	Procaine, benzocaine
Formulation:	Saturated aqueous solution of Furfural (2-Furaldehyde), slightly acidified with acetic acid.
Positive:	Red-violet
Stability:	Volatile. Should be kept refrigerated.

SCOTT'S

Intended Use:	Cocaine
Formulation:	A. 50 mL 2% cobalt thiocyanate 50 mL U.S.P. glycerine B. Concentrated hydrochloric acid C. Chloroform In test tube, add 5 drops A (if positive, will result in a blue precipitate), add 1 drop B (clear solution turns pink), add 3 to 4 drops C (blue color will transfer to chloroform layer).
Positive:	Pink-top layer/Blue-bottom layer
Stability:	Stable
Notes:	Some false positives.

SODIUM NITROPRUSSIDE

Intended Use: Secondary Amines (e.g. Methamphetamine or MDMA)

Formulation:
A. 0.5 g sodium nitroprusside dissolved in 50 mL water
1 mL acetaldehyde
B. 5 g sodium carbonate dissolved in 100 mL water

Add 1 drop of A, then 2 drops of B.

Positive: Blue, purple

Stability: Reagent A is volatile and should be refrigerated. Reagent A is blue when bad.

Notes: Very few false positives.

WEBER

Intended Use: Psilocyn

Formulation:
A. 0.01 g Fast Blue B
10 mL deionized water
B. Concentrated HCl

Add 2 drops of A, then 1 drop of B.

Positive: Red after addition of A and color change to blue after addition of B

Stability: Reagent A is unstable. Store in the freezer. If a freezer is not available, the reagent should be stored in the refrigerator and may be used until it no longer produces the expected results with the required primary standards as specified in DC-App IV-D. Do not store at room temperature. Expiration will be one year after the date of preparation, if the previously mentioned storage conditions are followed. Per QM-14, the frozen reagents will not be re-authenticated after one year.

Notes: A positive test result does not exclude psilocybin from being present.
A negative test result does not exclude psilocyn from being present.

WINTERGREEN

Intended Use: Cocaine

Formulation: Saturated potassium hydroxide or sodium hydroxide in methanol.

Note: Can also use 5% hydroxide solution.

Positive: Wintergreen odor

Stability: Stable

Notes: Water will interfere with the reaction.

ANION TEST: SILVER NITRATE

Intended Use:	Chloride ion, Iodide ion
Formulation:	1 g silver nitrate dissolved in 20 mL water
Positive:	White precipitate for chloride Yellow precipitate for iodide
Stability::	Stable

ANION TEST: BARIUM CHLORIDE

Intended Use:	Sulfate ion
Formulation:	10% barium chloride in water
Positive:	White precipitate
Stability:	Stable

ANION TEST: RING PRECIPITATION TEST

Intended Use:	Phencyclidine (PCP)
Formulation:	A. Sulfuric Acid: H_2O (4:1) B. Concentrated NH_4OH Add a sufficient amount of A to cover the sample. Then add drops of B along the sides of the test tube.
Positive:	Ring of Precipitate forms at the acid/base interface
Stability:	Stable
Notes:	Some false positives.

CHROMIC ACID

Intended Use:	GHB, GBL, 1,4-Butanediol
Formulation:	5M H_2SO_4 saturated with Chromic Oxide
Positive:	Blood Red-Brown
Stability:	Volatile, should be refrigerated.
Notes:	Can give false positive and false negatives.

FERRIC CHLORIDE

Intended Use:	GHB, GBL
Formulation:	Distilled H_2O saturated with $FeCl_3$
Positive:	Rust Red-Brown
Stability:	Keep refrigerated.
Notes:	May give false positives.

1% COBALT NITRATE IN EtOH SOLUTION

Intended Use:	GHB
Formulation:	1% Cobalt nitrate in an absolute ethanol solution
Positive:	Violet
Stability:	Stable
Notes:	May give false positives.

TOLUENE/SOLID COBALT THIOCYANATE

Intended Use:	GBL
Formulation:	Add approximately 1 mL of sample into a test tube followed by 0.5 mL of toluene. Vortex. Place a few crystals of solid cobalt thiocyanate in a spot well and add 3-5 drops of the toluene layer.
Positive:	Blue color developing upon drying
Stability:	Stable
Notes:	Running a blank is suggested, may give false positives.

INSTRUMENTATION

None

MINIMUM STANDARDS AND CONTROLS

The following conditions are to be met for the use of color tests or chemical tests (classical functional group tests):

1. A color test, if positive, may be used as a preliminary test for the identification of a controlled substance.

2. Color tests cannot be used as the preliminary test for a no scheduled substance found determination.

3. Notation of test done, and color or reaction produced will be recorded on the worksheet.

4. Stock bottles of color reagents are to be tested when prepared and as indicated in QM-14, using primary or secondary standards. Record retention will be followed per QM-14.

   Color reagents will be labeled according to QM-14, including a hazard label.

5.

PROCEDURE OR ANALYSIS

The color tests are conducted by transferring a small amount of substance to a non-reactive surface. Suggested surfaces are porcelain spot plates, disposable polyethylene spot plates, culture tubes or shell vials. The suggested number of drops of reagent in the proper order is added to the sample. Any observed color, absence of color, odor or effervescence is noted.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. The Drug Chemistry Training Manual, Section DC-IA-4.

2. Clarke, E. G. C. *Isolation and Identification of Drugs*, 2nd ed.; Pharmaceutical Press: London, England, 1986.

3. Feigl, F. *Spot Tests in Organic Analysis*, 7th ed. Elsevier Publishing: New York, NY, 1966; pp. 137-139, 251, 381-382.

4. Chemical Companies:

Sigma-Aldrich Chemical Company
P O Box 14508
St. Louis, MO 63178-4508
1-800-325-3010

GFS Chemicals
P O Box 245
Powell, OH 43065-0245
1-800-858-9682

Fisher Scientific
300 Industry Drive
Pittsburgh, PA 15275
1-800-766-7000

Avantor Performance Materials
3477 Corporate Parkway
Center Valley, PA 18034
1-855-282-6867

Alfa Aesar Chemical Company
A. Johnson Matthey Company
2 Radcliff Road
Tewksberry, MA 01876
1-800-343-0660

5. Garrett, A., Clemens, S., Gaskill, J., "The Weber Test: A Color Test for the Presence of Psilocin in Mushrooms," SWAFS Journal. 1993, Vol. 15, No. 1, pp 44-45.

6. Koppenhaver, D. J., "GHB Color Test," Microgram. 1997, Vol. XXX, No. 6, p. 130.

7. Morris, J., "Extraction of GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL)," Microgram. 1999, Vol. 8, pp. 215-221.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: THIN-LAYER CHROMATOGRAPHY (TLC)

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INTRODUCTION

Thin-Layer Chromatography (TLC) is a preliminary test. It can be used as a general screening test and the relative retention factor (Rf) and visualized colors can be compared to a known standard that is run with the sample to verify the results.

SAFETY CONSIDERATIONS

Standard Laboratory Practices. Both mobile phases and visualizers should be prepared in the hood. Also, any spraying of visualizers should be performed in the hood with the spray directed into a spray box or similar container.

PREPARATIONS

Unless otherwise noted, all chemicals should be of the highest grade attainable (ACS or reagent grade preferably) to avoid contamination problems and to ensure reactivity.

VISUALIZERS

Vanillin	1 gram vanillin in 20 mL sulfuric acid
KIPT	0.25 g Platinic Chloride 5 g Potassium Iodide Dilute to 100 mL with water
Acidified KIPT	5 mL concentrated Hydrochloric Acid is added to 100 mL KIPT
Ehrlich's	Ehrlich's color reagent diluted 1:1 with Ethanol (95%)
Fiorese's	Reagent A: 10 % Ferric Chloride in water Reagent B: 15% Potassium Iodide in water 1 mL concentrated Hydrochloric Acid Reagent C: Water (optional)
N,2,6	Reagent A: 1% Sodium Hydroxide in Methanol Reagent B: 0.5% of N,2,6-Trichloro-p-benzoquinoneimine (TCBI) in Chloroform
Fast Blue B salt	0.2 g in 10 mL of water
Fast Blue BB salt	0.2 g in 10 mL of water

MINIMUM STANDARDS AND CONTROLS

The following are requirements to meet the minimum standards and controls for Thin-Layer Chromatography:

I. Documentation

1. The solvent system and method of visualization will be documented on the worksheet (abbreviations are acceptable).

2. TLC chambers will be labeled with the solvent system, preparer's initials, and date prepared.

3. Reference standards (primary or secondary) will be run on the plate. The results will be indicated on the worksheet. The lot number or laboratory inventory number of the standard(s) must be documented in the case file.

4. All TLC visualizing reagents will be tested when prepared and as indicated in QM-14, using a primary or secondary standard. Labelling, including a hazard label, and record retention will be followed per QM-14.

5. Reviewable data such as a photo (color, if possible, otherwise accurately labeled) of the developed TLC plate shall be included in the case file.

6. Spot color development (if any) and relative retention factor calculations must be documented in the case file.

II. Comparisons

1. After the plate is deemed suitable for comparison (as delineated in DC-App II), a direct comparison shall be made between the sample and the included reference standard(s).

2. The relative retention factor shall be calculated for spots of target analytes that will be subject to a comparison. Measurements shall be taken (in centimeters) from the origin to the center of the spot. For instances where multiple samples of the same case/item number are run on the same plate and are referenced to a single standard, it is acceptable to document the relative retention factor of each extreme sample spot (i.e. the low and the high) instead of every individual spot.

3. A positive result or indication occurs when the relative retention ratio is between 0.85 and 1.15. If a chemical visualizer is used and a color is present, the spot(s) must exhibit a similar color reaction as the reference standard(s).

FORMULA FOR A RELATIVE RETENTION FACTOR CALCULATION

Rf of sample versus Rf of Reference Standard Run on the Same Plate ($=R_{st}$)

$$R_{st} = \frac{\text{Analyte distance}_{sample}}{\text{Analyte distance}_{standard}}$$

PROCEDURE OR ANALYSIS

Reference standard(s) must be run on the same plate as the unknown sample(s). Commercially prepared TLC plates (or HPTLC plates) with or without a fluorescent indicator will be used. The developing solvent is allowed to equilibrate in a sealed TLC chamber. (Note: For HPTLC, a smaller glass chamber is used since only 5 to 10 mL of solvent is needed.)

The reference standard(s) and unknown sample(s) are dissolved in a suitable, non-aqueous solvent and spotted on the origin above the solvent line. The plate is placed in the chamber and developed to the desired height. The plate is then air dried, visualized by ultraviolet (UV) light and/or sprayed with a selected visualizing agent. A positive test results if the relative retention ratio is within the acceptable range and, if visualized with a chemical visualizing agent, their colors are similar. These and other factors are noted.

EXAMPLES OF SOLVENT SYSTEMS

TYPE OF DRUG	DEVELOPING SYSTEM (Vol: Vol Ratio)
Cannabis	Hexane: Ether (4:1)
Cannabis, Salvinorin A	Hexane: Acetone (4:1)
General Screen	Methanol: Ammonia (95:5)
General Screen	CDEN (Chloroform, Dioxane, Ethyl Acetate, Ammonia) (25:60:10:5)
General Screen	Acetonitrile: Ammonia (95:5)
LSD	Acetone: Ammonia saturated chloroform (2:1)
LSD	Cyclohexane: Acetone: Diethyl Ether: Diethylamine (35:30:30:5)
Psilocyn/Psilocybin	n-Butanol: Acetic Acid: Water (2:1:1)
Psilocyn/Psilocybin	Methanol: Ammonia (95:5)
Salvinorin A	Hexane: Ethyl Acetate (1:1)
Steroids	Chloroform: Ethyl Acetate (4:1)

EXAMPLES OF VISUALIZERS

TYPE OF DRUG	VISUALIZER
General Screening	KIPT
	Fiorese's
Cannabis	Fast Blue B
	Fast Blue BB
	N,2,6-Trichloro-p-benzoquiononeimine (TCBI)
LSD	diluted Ehrlich's
Psilocyn/Psilocybin	diluted Ehrlich's
Salvinorin A	Vanillin
Steroids	Ethanol: Sulfuric Acid (4:1)

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. The Drug Chemistry Training Manual, Section DC-IA-5.

2. Touchstone, J., Dobbins, M. Practice of Thin-Layer Chromatography, 2nd ed., John Wiley and Sons: New York, NY, 1983, pp. 1-15, 103-136.

3. Clarke, E. G. C. Isolation and Identification of Drugs, 2nd ed.; Pharmaceutical Press: London, England, 1986, PP 146-147.

4. Kirchner, J. G. (1967). *Technique of Organic Chemistry* (Vol. XII). (E. W. Perry, Ed.) New York: Interscience Publishers, pp. 8.

5. Munro, Thomas A., Goetchius, Glen W.; Roth, Bryan L., Vortherms, Timothy A., Rizzacasa, Mark A. (2005), Autoxidation of Salvinorin A Under Basic Conditions, Journal of Organic Chemistry, Vol. 70, pp. 10057-10061.

6. Valdéz III, Leande, J, Chang, Hui-Ming, Visger, Daniel C., Koreeda Masato. (2001). Salvinorin C, a new neoclerodane diterpene from a bioactive fraction of the hallucinogen Mexican mint Salvia divinorum. Organic Letters, Vol. 3, No. 24, pp. 3935-3957.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: GAS CHROMATOGRAPHY WITH FLAME IONIZATION DETECTION (GC-FID)

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INTRODUCTION

Gas Chromatography with Flame Ionization Detection (GC-FID) can be used for qualitative and quantitative purposes or as a general screen to detect the presence of controlled substances. The instrument can be used to perform individual analysis or in conjunction with an autosampler for automated analysis. Comparison of retention time data generated via GC-FID is a preliminary test.

SAFETY CONSIDERATIONS

Standard Laboratory Practices.

PREPARATIONS

The samples should be dissolved in a suitable solvent, e.g., chloroform, methanol, etc.

INSTRUMENTATION

Gas Chromatograph with Flame Ionization Detector with or without autosampler

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

The following are requirements to meet the minimum standards and controls for GC-FID:

I. Documentation

1. A copy of the chromatogram(s) will be preserved in the case file.

2. The worksheet will indicate the result of the GC-FID and all chromatograms must be referenced to the worksheet (excluding standards). The chromatograms must be traceable to the GC-FID from which they were generated. Data that is NOT suitable for comparison must be able to be distinguished from data that is suitable for comparison. This can be accomplished by associating samples with their corresponding chromatograms and results in the "Notes" panel in LIMS or through annotations on the included data.

3. The gas chromatography conditions, including the carrier gas, column, injection temperature, injection volume, split ratio, oven temperatures, oven ramps, and hold times, will be recorded in the case file. Any additional modifications to the method (e.g. turning the detector off/on including post run holds, pressure ramps, etc.) must be noted in the case file.

4. The case number, analyst's initials and date will be recorded on all copies. Item number is optional on blanks and reference standards but required on all other copies.

II. Screening Parameters

1. For a general screening run, the injection volume will be between 1 and 3 microliters and the split ratio will be between 20:1 and 50:1 (for Shimadzu brand instruments and Agilent 8890 instruments, the injection volume may be as low as 0.5 microliter and the split may be as high as 200:1).
    a. "Weak" methods that alter the split ratio, injection volume, or that employ manual injections to allow more sample onto the column are acceptable for use after a general screening run has been attempted.
    b. When appropriate, it is acceptable to use a weak method for an initial screening (e.g. residue or small sample amount), however, this should not be routine and will be documented on the worksheet.
    c. Methods tailored for the identification of a particular drug (or class of drugs) are exempt from these requirements.

III. Comparisons

1. Any qualitative comparison(s) will be made to in-house standards after the data has been assessed for suitability (see DC-App II).
    a. The standards will be run on the same instrument and under the same conditions, including the column, carrier gas, oven temperatures, oven ramps, and oven hold times, as well as any other method modifications affecting retention time. (Injection parameters are excluded as they relate to sample introduction, not separation.)
    b. For retention time comparisons, data shall not be more than six months old.
    c. In order to be considered a positive result or indication, the retention time of the apex of the sample peak must be within +/- 1% of the retention time of the apex of the appropriate reference standard.
    d. The standard for comparison (including the lot number or laboratory inventory number) must be included in the case file.

IV. Function Checks / Positive Controls

1. A logbook (or online equivalent) will be kept for the instrument to record maintenance, function checks, positive controls, standards for comparison, and the column installation date.
    a. To insure the instrument is working properly, a positive control will be run monthly or more frequently if indicated by instrument performance or before operation if not used on a routine basis. For the purposes of GC-FID, the positive control is one or more reference materials run under the same conditions (isothermally or using a temperature program) on the same method on a routine basis. Before accepting a positive control, the chromatogram must be compared to a previously accepted control or to reference material and examined for peak shape, height, and retention time reproducibility.
    b. A program to be used as a general drug screen or unknown run will be checked by running low and high boiling standards. A general screening run shall

demonstrate the ability to detect both GHB or GBL and Buprenorphine or Trazodone. This should occur monthly or before operation if the instrument is not used on a routine basis. If multiple screening methods vary only by their injection parameters and / or final hold time, only one needs to be verified. Injection parameters are not crucial, and the shortest final hold time should be chosen for verification.

c. After the installation of a column and/or column maintenance (i.e. column clipping), verification of the general screen method will be completed to demonstrate the instrument's ability to detect a range of substances. If an instrument is used for a specific purpose that does not include general drug screening, a positive control consistent with a routine function check will be used to verify if the instrument is working properly.
d. After detector maintenance, a positive control consistent with a routine function check will be used to verify the instrument is working properly.

PROCEDURE OR ANALYSIS

SAMPLE DRUG PROGRAM

The following method is an example of a general drug screen. It includes low boilers (GHB, GBL) and high boilers (Buprenorphine, Trazodone).

Temperatures/Degrees Celsius

Injector	250
Detector	280
Oven	70 for 1 minute, ramp to 280 at 30/minute, hold at 280 for 10 minutes

Column

12 M, 5% Phenyl Methyl Siloxane ID 0.2 mm, film thickness 0.33 micrometer

Carrier Gas

Nitrogen, Hydrogen, or Helium

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. The Drug Chemistry Training Manual, Section DC-IA-7.
2. Grob, R., *Modern Practice of Gas Chromatography*, 2nd ed., John Wiley and Sons: New York, NY, 1985, pp. 159-176.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

PROCEDURE: TRANSMISSION AND INTERNAL REFLECTANCE

Reviewed by:

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Forensic Scientist Adrienne A. Hirsch, Chairperson
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Approved by:

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Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

Fourier Transform Infrared Spectroscopy (FTIR) can be used as a screening procedure or, most commonly, as a tool for positive structural identification of a compound (confirmatory testing technique). FTIR data may be acquired by multiple methods. Two of the methods employed by ISP include transmission and internal reflection. Both methods often require purification of the sample prior to analysis.

Transmission spectra are produced by measuring the energy transmitted through a sample when a single-path infrared beam passes through it. Sample preparation may require chemical extraction in addition to pellet preparation.

For FTIR using internal reflection, a multiple-path infrared beam is passed through an internal reflecting element (IRE) with a high refractive index at a certain angle. The most common IRE is a diamond, and acquisition is usually via an attenuated total reflection (ATR) instrument accessory. The internal reflection creates an evanescent wave which extends above the crystal IRE. When a sample is held in contact with the IRE, the sample absorbs energy and the evanescent wave is altered or attenuated. This attenuated energy is passed back into the infrared beam. A detector measures the changes in the beam and produces an infrared spectrum.

Identifications made using FTIR rely on comparisons of peak positions and relative peak intensities between sample and standard spectra. This is true whether data is acquired via transmission or internal reflection methods. While transmission and internal reflection spectra of the same substance will show distinct similarities and may provide useful information as to the identification of a specific substance, care must be taken to ensure that identifications are made based on comparisons of spectra acquired in the same manner (i.e. transmission or internal reflection).

RELATED PROCEDURES

Extractions (Appendix III)

SAFETY CONSIDERATIONS

FTIRs use a laser beam to align the mirrors during the operation of the instrument. Do not look directly into the beam as damage to the eyes can result.

PREPARATIONS

In some instances, such as when an ATR accessory is used, there is no sample preparation required. The sample may be used directly (run neat) or extracted. However, when a pellet is required for transmission spectral acquisition, the sample must be pressed into an IR-transparent pellet, using

IR grade Potassium Bromide and ground with a mortar and pestle.

Potassium Bromide Note: An IR of the KBr will be run when the new bottle is opened. The bottle will be marked with the date opened and initialed. The IR scan will be preserved until the KBr is consumed in analysis.

INSTRUMENTATION

Fourier Transform Infrared Spectrophotometer with
A Pellet Press OR
An Attenuated Total Reflection Accessory

Reference: Operator's Manuals

MINIMUM STANDARDS AND CONTROLS

The following are requirements to meet the minimum standards and controls for Infrared Spectroscopy:

I. Documentation

1. A copy of the IR spectrum will be included in the case file. At least two peaks will be peak-picked and labeled with the wavenumber of the apex. These peaks should be within the 2000-1300 cm^{-1} region, whenever possible. The peaks selected for labeling shall correlate directly to the peaks used for comparison to a reference standard, which will be noted in the case file. In addition, if using an ATR accessory, a blank will be run and included in the case file. An ATR accessory requires a blank to be run before every sample.

2. The IR collection parameters (number of scans, resolution) as well as the case number, item number, analyst's initials, and date will be recorded on spectra. Item number is optional on standards. The optical isomer (where applicable) and the salt form of the standard used for comparison will be recorded.

3. The worksheet will indicate the result of the IR and any additional spectra will be referenced to the worksheet (excluding standards). Spectra shall be traceable to the IR from which they were generated. Data that is NOT suitable for comparison, but is included in the case file, must be distinguishable from data that is suitable for comparison. This can be accomplished by associating samples with their corresponding spectra and results in the "Notes" panel in LIMS or through annotations on the included spectra.

4. If an acquisition method other than the ATR accessory is used, it shall be indicated in the work notes.

II. Screening Parameters

1. The following is an example of recommended method parameters:

Number of scans: 32 to 36*
*internal reflectance scans should be a square number
Resolution: 4 cm^{-1}
Gain: 1
Apodization: Triangular
Starting wavenumber: 4000 cm^{-1}
Ending wavenumber: 500 cm^{-1}

III. Comparisons

1. Any qualitative comparison(s) will be made to in-house standards after the data has been assessed for suitability (see DC-App II).
2. To make an identification, the spectrum must be compared to an in-house, peak- picked primary standard run on the same instrument and under the same conditions. For FTIR, this includes number of scans, scan range, resolution, and acquisition method (pellet or ATR).
3. A positive identification may be made when:
    i. Sample and standard spectra exhibit general pattern correspondence of peaks and relative intensities, and
    ii. At least two peaks are compared between the sample and the reference standard and are within +/- 1 cm^{-1} of each other. These peaks should be within the 2000-1300 cm^{-1} region whenever possible. When two peaks are not present in this region, then the most abundant peaks will be compared. Corresponding peak wavenumbers will be labeled on both the sample and the reference standard spectra. [NOTE: transmission and internal reflectance methods should produce *similar* spectra, but corresponding wavenumbers may differ by more than +/- 1 cm^{-1}. If a reference standard is available that was acquired by a different mode than the sample spectrum, the data may still be used to *support* a conclusion, given the presence of relevant data from a separate confirmatory testing technique.]
4. The reference standard used for comparison (including the lot number or laboratory inventory number) must be included in the case file, and the wavenumbers of the two peaks used for the comparison will be documented.
5. In the event that a primary, in-house spectrum is not available, an identification maybe made using two peer reviewed reference sources, ie. IDDA or Pfleger, provided the following is true:

i. Both sources are of independent and known origin, AND
ii. Sample and both reference spectra exhibit overall pattern similarities, AND
iii. On the sample and at least one of the standard reference spectra, exact wavenumbers of at least 2 peaks are identified, within the 2000-1300 cm^{-1} whenever possible, and are within +/- 1 cm^{-1} of each other, AND
iv. Reference standards were obtained by the same acquisition method as the sample (e.g. samples analyzed via ATR must be compared to standards run via ATR), AND
v. Images of the sources used for comparison shall be included in the case file.

IV. Function Checks/Positive Controls

1. A polystyrene standard or an internal function check mechanism (e.g. Valpro) will be run at least once a month on both the FTIR and ATR (when present), or more frequently if indicated by instrument performance. If the instrument is not used on a routine basis, the polystyrene standard or internal function check may be completed before use. The reports of the internal function check mechanism must pass all aspects of the reported tests as defined by the manufacturer and will be compared to previous run reports. A source voltage value, the IR peak to peak (P2P) value, and the P2P value for the ATR will be recorded and maintained in the instrument logbook (or online equivalent). For a single bounce diamond ATR accessory, the P2P with the ATR installed should be at least 10% of the FTIR P2P. If it is not, an alignment may need to be performed.
2. A positive control shall be run following any function checks. The **same** traceable reference material will be run monthly or as needed under the same instrumental conditions. A traceable polystyrene reference or drug standard will be used as the positive control. These control spectra will be peak-picked and compared to previous peak-picked spectra, and maintained in the instrument logbook (or online equivalent). Deviations of greater than +/- 1 cm^{-1} of prominent peaks in the 2000-1300 cm-1 region from the prior function check will need to be considered.

PROCEDURE OR ANALYSIS

I. FTIR using internal reflectance (ATR accessory)

1. Background Spectrum
   i. The ATR attachment should be cleaned, and a new background collected prior to use. The background is to be acquired with the arm in the up position.
   ii. It is advisable to collect a background spectrum every 5-10 minutes to minimize the impact of environmental factors on analytical spectra.
2. Blank Spectrum
   i. Prior to each sample, a blank spectrum should be run with the arm down. It will be included in the working file and will be noted on the worksheet.

ii. The analyst will ensure that the ATR attachment is clean. If the blank has any unusual peaks, the ATR attachment must be re-cleaned and the blank run again.

3. Sample Spectrum
   i. A sample is placed on the diamond mount area. The clamp containing the appropriate anvil is secured **only to the point that good contact is maintained** between the sample and the diamond.
   ii. When working with a liquid, it should be dropped onto the diamond mount and allowed to evaporate completely so that crystals may form directly on the diamond before clamping the anvil.
   iii. The resulting spectra are compared to standards obtained in the same manner.

II. FTIR using transmission (pellet)

1. A small amount of the sample is mixed with KBr using a mortar and pestle. After the two have been sufficiently mixed, the mixture is placed in the pellet die and a KBr pellet is made. (The size of the pellet is optional). If necessary, a background spectrum of KBr should be taken and stored in the computer. The sample pellet is placed on the IR and a spectrum of the substance is obtained.

REPORT WORDING

See Appendix I – Report Wording

REFERENCES

1. The Drug Chemistry Training Manual, Section DC-IA-8.

2. Graf, R.T.; Koenig, J.L.; and Ishida, J.; In Fourier Transform Infrared Characterization of Polymers; H. Ishida, Ed.; Plenum: New York, 1987.

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ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: GAS CHROMATOGRAPHY - MASS SPECTROMETRY (GC-MS)

Reviewed by:

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Drug Chemistry Command Advisory Board

Approved by:

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Forensic Sciences Command

INTRODUCTION

When a gas chromatograph is coupled with a mass selective detector, it gives rise to the hyphenated technique known as GC-MS, or gas chromatography-mass spectrometry. GC-MS can be used for screening purposes, however, its most common use is for structural information from the mass spectrometer for the positive identification of a compound. The instrument can be used with an automated sampler and for quantitation.

The qualitative analysis of unknown substances by GC-MS is accomplished by direct comparison with a standard spectrum for an identification. The mass spectrum must be interpreted. This does not mean that all the fragments must be assigned but rather the chemist must determine if the unknown and the standard are the same substance. This is done by comparing the mass of each fragment and its relative intensity in the unknown to the standard. Retention time comparisons from GC-MS can also be used as preliminary testing. When retention time data is used in conjunction with mass spectral data, a GC-MS run is still considered a single test for the purposes of meeting the minimum requirement of two tests.

The computer is used to acquire data in a form which can be used by the chemist. It should not alter the fundamental data.

SAFETY CONSIDERATIONS

Standard Laboratory Practices.

PREPARATIONS

The sample will usually be dissolved in a suitable solvent prior to introduction into the GC-MS. Examples include but are not limited to:

Methanol
Chloroform

INSTRUMENTATION

Gas Chromatograph with Mass Selective Detector with or without autosampler

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

The following are requirements to meet minimum standards and controls for gas chromatography - mass spectrometry:

I. Documentation

1. A copy of the spectrum and the entire total ion chromatogram will be included in the case file.
   a. If there are no peaks indicating the possible presence of an analyte in an unknown sample, i.e. the unknown sample TIC appears similar to a blank, a spectrum does not need to be included.

2. The identification of a compound must be based on visual comparison of the fragmentation pattern to that of a reference standard.

   a. Subtractions of background noise should not change the fundamental data, and therefore need not be recorded on the worksheet.
   b. Subtractions of a separate compound require the original, subtracted and resultant spectra to be documented in the case file.
      i. If multiple units have a similar original spectrum requiring a similar subtracted spectrum for each sample, one original and one subtracted spectrum from a single sample can be used for documentation.
         1. Spectra not included will be documented in the case file. For example, "Original and subtracted spectra included for 1.1, similar subtractions completed for 1.2-1.10, spectra not included".
         2. Resultant spectra for each unit will be included.

3. The instrumental conditions, including carrier gas, column, injection temperature, injection volume, split ratio, oven temperature, oven ramps, oven hold times, solvent delay, auxiliary or mass selective detector (MSD) interface temperature, scan range, and tune file or tune type (e.g. atune, stune, etc.) will be recorded in the case file. Any additional modifications to the method (e.g. turning the detector off/on including post run holds, pressure ramps, etc.), not included on the footer, must be noted in the case file.

4. The case number, analyst's initials, item number, and date will be recorded on all copies. Item number is optional on standards. (Per the blank policy in Appendix IV-A, item number is also optional on blank chromatograms/spectra.)

5. The worksheet will indicate mass spectrometer results and all spectra must be referenced to the worksheet (excluding standards). The spectra will be traceable to the GC-MS from which they were generated. Data that is NOT suitable for comparison must be distinguishable from data that is suitable for comparison. This can be accomplished by associating samples with their corresponding spectra and results in the "Notes" panel in LIMS or through annotations on the included spectra.

II. Screening Parameters

1. For a general screening run, the injection volume will be between 1 and 3 microliters and the split ratio will be between 20:1 and 50:1 (for Shimadzu brand instruments and Agilent 8890/5977 instruments, the injection volume may be as low as 0.5 microliter and the split may be as high as 100:1).
    a. "Weak" methods that alter the split ratio, injection volume, or that employ manual injections to allow more sample onto the column are acceptable for use after a general screening run as been attempted.
    b. When appropriate, it is acceptable to use a weak method for an initial screening (e.g. residue or small sample amount), however, this should not be routine and will be documented on the worksheet.
    c. Methods tailored for the identification of a particular drug (or class of drugs) are exempt from these requirements.

III. Comparisons

1. The mass spectrum shall be compared to an in-house standard, either primary or secondary, after the data has been assessed for suitability (see DC-App II).
    a. The standard must be run on the same instrument and under the same mass spectral conditions. This includes auxiliary or MSD interface temperature, scan range, and tune file or tune type. (Solvent delay is not required as it relates to turning the detector off/on.)
    b. The standard for comparison (including the lot number or laboratory inventory number) must be included in the case file.
    c. For a qualitative mass spectral comparison, the age of the spectrum of the in-house reference standard is not crucial if the analytical conditions meet the criteria in item (a) above, and the instrument has met the requirements of function checks and positive controls.
    d. An identification shall only result when a preponderance of ions across the spectrum exhibit general correspondence of abundances between ions of corresponding nominal *m/z* values.
        a. The sample and the reference standard shall exhibit the same base peak, unless variations have been documented by the laboratory.
        b. The sample and reference standard shall exhibit the same molecular ion, when observed, unless variations have been documented by the laboratory.
        c. Isotopic ion distributions observed in the reference spectrum shall be present in the sample spectrum.
    e. In the case that a standard is unavailable, an identification may be made using two appropriate literature sources or external standard references, provided the sources are evaluated according to parameters outlined in DC-App IV-A. Care must be taken to verify that mass spectral data coupled to other available testing is sufficient to conclusively identify the substance in question. Additionally, the following conditions must be met:
        a. The sources used for comparison must be of independent origin, and these shall be documented in the case file,
        b. Both spectra shall be of sufficient abundance to allow for meaningful comparisons,

c. Both shall exhibit the same base peak and molecular ion, when one is observed,
d. Both shall exhibit general correspondence in relative abundance of a majority of ions across the spectra, and
e. Both shall be included in the case file.

2. If GC-MS data is used to compare retention times, this comparison will be clearly documented. It is not required to utilize this information unless it is necessary to support a specific conclusion. (Examples of useful retention time comparisons include, but are not limited to, isomer differentiation such as between pseudoephedrine and ephedrine, or the positional isomers of methylethcathinone.)
   a. If no retention time comparison is noted, it will be understood that the retention time data from the coupled technique was not considered.
   b. For retention time comparisons, the standard for comparison must be run on the same instrument under the same conditions. This includes the column, carrier gas, oven temperatures, oven ramps, oven hold times, auxiliary or MSD interface temperature, scan range, and tune file or tune type, as well as any other method modifications affecting retention time. (Injection parameters and solvent delay are excluded as they relate to sample introduction into the system, not separation.)
   c. Criteria for reference standard expirations, positive test results, and reference standard documentation specifically associated with retention time data can be found in Section DC-D-1/III.

IV. Function Checks / Positive Controls

1. The function check for mass spectrometry utilizes Perfluorotributylamine (PFTBA) as a reference sample to tune the mass spectrometer. The following ranges are used to qualify the function check:

   - Mass assignments of 69, 219, 502 are +/-0.2 amu.
   - Isotopic ratio of 70 to 69 falls in the 0.5 – 1.6% range
   - Isotopic ratio of 220 to 219 falls in the 3.2 – 5.4% range
   - Isotopic ratio of 503 to 502 falls in the 6.5-13.5% range

2. A logbook (or online equivalent) will be kept for the instrument to record maintenance, function checks, positive controls, and standards for comparison.
   a. An autotune will be performed monthly or more frequently if indicated by instrument performance or usage, or before operation if not used on a routine basis. A record of the tune will be retained.
   b. A positive control will be run after every autotune. For the purposes of mass spectrometry, the positive control is one or more reference materials run under the same conditions, on the same method on a routine basis. Before accepting a positive control, it must be compared to a previously accepted control or reference material.

c. A program to be used as a general drug screen or unknown run will be checked by running low and high boiling standards. A general screening run shall demonstrate the ability to detect both GHB or GBL and Buprenorphine or Trazodone. This should occur monthly or before operation if the instrument is not used on a routine basis. If multiple screening methods vary only by their injection parameters and / or final hold time, only one needs to be verified. Injection parameters are not crucial, and the shortest final hold time should be chosen.
d. After the installation of a column and/or column maintenance, verification will be completed to demonstrate the instrument's ability to detect a range of substances. If an instrument is used for a specific purpose that does not include general drug screening, a positive control consistent with a routine function check will be used to verify if the instrument is working properly.
e. After mass spectrometer maintenance (i.e. source cleaning, filament switch/replacement, pump maintenance, etc.), a function check including autotune and positive control analysis will be completed.

PROCEDURE OR ANALYSIS

SAMPLE DRUG PROGRAM

The following method is an example of a general drug screen. It detects low boilers (GHB,GBL) and high boilers (Buprenorphine, Trazodone).

Temperatures (Degrees Celsius)

Inlet:	250
Auxiliary or MSD interface:	280
Oven:	70 for 0 minutes, ramp to 300 at 30/minute, hold at 300 for 7.3333 minutes

Injection Parameters

Injection Volume:	1.0 µL
Split Ratio:	30:1

Column

30 meter, 100% dimethyl polysiloxane ID 0.25 millimeter, film thickness 0.25 micrometer

Carrier Gas

Hydrogen or Helium

Detector Parameters

Solvent Delay:	1.0 min

Scan Range: 40 – 550 amu
Tune file/Tune type: stune.u

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. The Drug Chemistry Training Manual, Section DC-IA-9.
2. Watson, J. T., *Introduction to Mass Spectrometry*, Raven Press, NY, 1985; pp. 1-16, 75-93, 121-143, 153-165 189-295.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: SAMPLING PLAN

Reviewed by:

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Forensic Scientist Adrienne A. Hirsch, Chairperson
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Approved by:

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INTRODUCTION

When multiple unit cases are submitted for analysis, it may be appropriate to employ a statistically based sampling plan. In addition, the weight or number of tablets may need to be extrapolated. This section will outline Hypergeometric Sampling, Bayesian Sampling, and the Determination of Weight or Number of Units. The calculated number of units must be individually tested as to meet the Minimum Standards and Controls for analysis.

SAFETY CONSIDERATIONS

No additional safety considerations.

PREPARATIONS

No additional preparations are necessary.

INSTRUMENTATION

ENFSI Sampling Software (Second Edition, dated July 2017)

DEFINITIONS

Bias:	Having a preference or tendency to favor.
Consistent:	The same in physical appearance, markings, color, size.
Dosage:	Distinct segregated items that are intended for individual consumption.
Homogenous:	Uniform in physical appearance (size, color, consistency, markings, etc.)
Negative:	Having composition that strays in comparison to other items tested.
Positive:	Having composition that yields consistent results with all other items tested, whether the substance contained is controlled or not.
Random Sample:	Selection of items to be tested without bias.
Representative Sample:	A small sample portion that is characteristic of the whole population and is sufficient for testing.
Sampling:	Selecting a representative portion of a population to make a determination about the whole.
Test Portion:	The number of units determined from the sampling plan that is set aside for analysis.
Unit:	A single individual object/member of a population

MINIMUM STANDARDS AND CONTROLS

1. The case file and report will reflect the sampling plan used.

2. The sampling plan should maintain a confidence level of at least 95%.

3. A copy of the results of the sampling plan will be included in the case file. When employed, the results of a random number generator and/or weight estimation program will also be included.

PROCEDURE OR ANALYSIS

1. The population will be determined to be visually homogenous.

2. The worksheet will reflect the sampling plan used and a copy of the results of the calculations will be included in the case file.

3. Typically, with Hypergeometric Sampling, 90% of a population will be called. Adjusting the percentage of the population analyzed is at the discretion of the analyst in order to reach the highest attainable sentencing class. Utilizing Bayesian Sampling, the entire population will be called. In all cases, a confidence level of at least 95% will apply.

4. The units to be tested shall be randomly selected from the total population. A random number generator may be used.

5. Each unit in the test portion will be analyzed individually according to the testing requirements for that particular type of evidence (e.g., powders, tablets, plant material, etc.).

6. If a negative result is obtained, the sampling plan will be aborted and testing the appropriate number of units necessary for the charge will be completed.

REPORT WORDING

Clarification will be made in the description for extrapolated/estimated weights and/or number of units. (See H1-C)

The analyst will add a remarks line on the report to indicate the sampling plan utilized including the percentage of the population accounted for and the confidence level.

REFERENCES

1. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Representative Drug Sampling; 2003.

2. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Sampling of Illicit Drugs for Qualitative Analysis; Second Edition; 2016.

3. Coulson, S.A.; Coxon, A.; and Buckleton, J. “How Many Samples from a Drug Seizure Need to be Analyzed?” Jour. For. Sci. 2001; 46(6):1456-1461.

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11. Drug Chemistry Training Manual – Section DC-IA-17.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: SAMPLING PLAN

PROCEDURE: HYPERGEOMETRIC SAMPLING

Reviewed by:

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INTRODUCTION

Hypergeometric Sampling is a mathematical probability calculation used to make a determination on a population, based on testing a representative sample of that population. The population to be called positive and the number to be tested can be determined from the ENFSI software program. Hypergeometric Sampling will always have a "Not Analyzed" portion of the population. It is recommended that the Hypergeometric plan is applied to populations of at least 35 units. This plan may not be appropriate for situations where the estimated weight or the total population is near or at a charging limit.

INSTRUMENTATION

ENFSI Sampling Software (Second Edition, dated July 2017)

PROCEDURE

1. The population will be determined to be visually homogenous. Remove any units that are not homogenous to the population and test each differing population appropriately.

2. Utilizing the ENFSI program, enter the total number of units in the population.

3. Enter the portion of the population to be called positive as a decimal. (e.g. 90% entered as 0.90)

4. Enter "0" for the number of negatives. (If a negative result is obtained, the sampling plan will be aborted.)

5. Enter a percent confidence level as a decimal. (e.g. 95% is entered as 0.95)

6. Attach the Calculation Parameter Table and Confidence Level vs. Sample Size graph (or "yes/no" table up to the number of units required to fit confidence level criteria in place of the graph) to the analytical notes packet. (SOFTWARE NOTE: Do not include all pages from the program.)

7. Test the appropriate number of units as determined by the calculations.

8. The report will reflect an Item (#) being the amount called positive and an Item (#) being a "Not Analyzed" portion.

REPORT WORDING

The following is an example of the report wording for use of hypergeometric sampling in a case. Remember, when using the Hypergeometric sampling plan, the call is the portion of the population (e.g. 90%) set as positive in the sampling calculation.

Item 1-1	21.2 grams of 270 tablets	3,4-Methylenedioxymethamphetamine (MDMA)
Item 1-2	2.3 grams of 30 tablets	Not Analyzed

REMARKS: The Hypergeometric sampling plan was utilized to analyze Item 1, accounting for 90% of the population at a 95% (or higher) confidence level.

REFERENCES

1. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Representative Drug Sampling; 2003.

2. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Sampling of Illicit Drugs for Qualitative Analysis; Second Edition; 2016.

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7. Drug Chemistry Training Manual – Section DC-IA-17.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: SAMPLING PLAN

PROCEDURE: BAYESIAN SAMPLING

Reviewed by:

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Forensic Sciences Command

INTRODUCTION

The Bayesian sampling plan is based on Bayes' Theorem. It is a statistically based sampling plan with a scientifically subjective conclusion drawn by the tendency of homogenous populations to test consistently. This experience and mathematics allow an analyst to test a representative sample of the population as determined from the ENFSI program (hypergeometric mathematics) and draw a conclusion for the entire population. A limit of 90% of the population at a 95% confidence level was chosen from the Coulson, et al. and the Frank, et al. articles. It is recommended that this approach is applied to a population of at least 50 units.

INSTRUMENTATION

ENFSI Sampling Software (Second Edition, dated July 2017)

PROCEDURE

1. The population will be determined to be visually homogenous. Remove any units that are not homogenous to the population and test each differing population appropriately.

2. Utilizing the ENFSI program, enter the total number of units in the population.

3. Enter a value of "1" for a and b if there is no prior information known. (This is the most conservative approach and strongly recommended. Those who are both comfortable in statistics and have an understanding of a and b can utilize a more aggressive approach and enter appropriate values for a and b.)

4. Enter 90% (as a decimal) as the portion of the population to be called positive. (e.g. 90% entered as 0.90.)

5. Enter "0" for the number of negatives. (If a negative result is obtained, the sampling method will be aborted.)

6. Enter a percent confidence level as a decimal. (e.g. 95% is entered as 0.95)

7. Attach the Calculation Parameter Table and Confidence Level vs. Sample Size graph (or "yes/no" table up to the number of units required to fit confidence level criteria in place of the graph) to the analytical notes packet. (SOFTWARE NOTE: Do not include all pages from the program.)

8. Test the appropriate number of units as determined by the calculations.

9. The conclusions will represent the total population.

REPORT WORDING

The following is an example of the report wording for use of Bayesian sampling in a case. Remember, when using the Bayesian sampling plan, the entire population is called positive based on the homogeneity and the experience of the analyst.

Item 1 21.0 grams of 1602 tablets 3,4-Methylenedioxymethamphetamine (MDMA)
REMARKS: The Bayesian sampling plan was utilized to analyze Item 1, accounting for 100% of the population at a 95% (or higher) confidence level.

REFERENCES

1. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Representative Drug Sampling; 2003.

2. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Sampling of Illicit Drugs for Qualitative Analysis; Second Edition; 2016.

3. Coulson, S.A.; Coxon, A.; and Buckleton, J. "How Many Samples from a Drug Seizure Need to be Analyzed?" Jour. For. Sci. 2001; 46(6):1456-1461.

4. Aitken, C. G. G. "Sampling – How Big a Sample?" Jour. For. Sci. 1999; 44(4):750-760.

5. Frank, R.S.; Hinkley, S.W.; and Hoffman, C.G. "Representative Sampling of Drug Seizures in Multiple Containers" Jour. For. Sci. 1991;36(2):350-357.

6. Joyce, James, "Bayes' Theorem", *The Stanford Encyclopedia of Philosophy* (Fall 2008 Edition), Edward N. Zalta (ed.), URL = <http://plato.stanford.edu/archives/fall2008/entries/bayes-theorem/>.

7. Drug Chemistry Training Manual – Section DC-IA-17.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: SAMPLING PLAN

PROCEDURE: DETERMINATION OF WEIGHT OR NUMBER OF UNITS

Reviewed by:

_____

Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

There are cases that may be submitted where sampling is the preferred method of analysis. In these cases, it may be necessary to estimate the total weight or the total number of units (especially tablets) in a population. The extrapolated weight will be determined using individual unit weights, the mean, and the standard deviation. The extrapolated number of units can be determined using the total population weight and the weight of a known number of units. The use of these estimates/extrapolations will be made clear in the report.

SAFETY CONSIDERATIONS

Standard Laboratory Practices

PREPARATIONS

No additional preparations are necessary.

INSTRUMENTATION

ENFSI Sampling Software (Second Edition, dated July 2017)

MINIMUM STANDARDS and CONTROLS

1. The calculated values for the mean and standard deviation will be noted on the worksheet/work notes.

2. A copy of the estimation of weight spreadsheet will be included in the case file.

3. The report will reflect that the weight is calculated, or the number of units estimated, as applicable to the case.

PROCEDURE – DETERMINATION OF THE NUMBER OF UNITS

The total number of units in the population (e.g. tablets) may be manually counted. If the population is large, the population number may be determined using the following:

1. The weight of the total population will be recorded.

2. The weight of a known quantity of units will be recorded.

3. The weight of the known quantity divided by the number of units in that quantity will be done.

4. The results of the division will be divided into the total weight of the population and the result will be the extrapolated number of units.

$$\text{Extrapolated Number of Units} = \frac{\text{Weight of population}}{\text{(Weight of known number of units/Known number of units)}}$$

An example. . .

$$X = \frac{\text{15.7 grams total weight of population}}{\text{1.4 grams / 10 units from population}}$$

$$X = \text{112 estimated units}$$

PROCEDURE – DETERMINATION OF WEIGHT (ENFSI software)

1. The number of units to be tested (sample size), as determined by the sampling plan (Hypergeometric or Bayesian), will be individually weighed. These weights will be recorded in an Excel spreadsheet.

2. The mean (or average, μ) of the sample size weights will be calculated using Excel (AVERAGE).

3. The standard deviation of the sample size weights will be calculated using Excel (STDEV.P).

4. Enter the values in the ENFSI software Estimation of Weight panel.

    a. Enter the confidence level (0.95)
    b. Enter the population (90% of the total population)
    c. Enter the sample size
    d. Enter the mean
    e. Enter the standard deviation
    f. Enter the number of negatives (0)
    g. Enter the weighing balance uncertainty (calculated using the average weight of the sample size in the appropriate Measurement Uncertainty Worksheet)
    h. Report the calculated weight of the appropriate number of units (90% of the population for Hypergeometric or the total population for Bayesian), ensuring the range is sufficiently above the necessary weight limit

    ** The total population is not necessarily the same (N) value that was used in the sampling plan calculation. The total population (N) is the total population of that item (sub-item). For one calculation, (N_1) would represent the positive population, for the next calculation (N_2) would represent the not analyzed portion. [Where the called population (N_1) + the not analyzed portion (N_2) = the total population (N).]

    For example:

There are 1000 packets of suspected heroin. When utilizing the Bayesian sampling plan, the total population is 1000 for determination of weight. If the Hypergeometric sampling plan was utilized at 90% of the population, the determination of weight would then be performed twice: the first time with a total population (N) of 900, and the second time with the remaining population (N) of 100. This may also be applied by using the total population of 1000, then the called population of 900, and by difference determine, the calculated weight of the 100 packets of the remaining population.

REPORT WORDING

The following are examples of descriptors that clarify that the number of units or the weight was extrapolated/estimated. When statistics are used for weight determination, use the word "calculated." For unit number determinations use the word "estimated." *These are only examples and not an exhaustive list. Remarks lines are necessary when a sampling plan is utilized.*

Determined number of units with known weights using Bayesian Sampling. . .

Item 1	218.9 grams of an estimated 721 tablets	3,4-MDMA

REMARKS: The Bayesian sampling plan was utilized to analyze Item 1, accounting for 100% of the population at a 95% (or higher) confidence level.

Determined number of units with known weights using Hypergeometric Sampling. . .

Item 1-1	12.3 grams of an estimated 1620 paper squares	LSD
Item 1-2	1.3 grams of an estimated 180 paper squares	Not Analyzed

REMARKS: The Hypergeometric sampling plan was utilized to analyze Item 1, accounting for 90% of the population at a 95% (or higher) confidence level.

Calculated weight with known units using Bayesian Sampling . . .

Item 1	412 vials of liquid weighing a calculated 604.1 grams	PCP

REMARKS: The Bayesian sampling plan was utilized to analyze Item 1, accounting for 100% of the population at a 95% (or higher) confidence level.

Calculated weight with known units using Hypergeometric Sampling . . .

Item 1-1	900 packets of powder weighing a calculated 109.2 grams	Heroin
Item 1-2	100 packets of powder weighing a calculated 12.1 grams	Not Analyzed

REMARKS: The Hypergeometric sampling plan was utilized to analyze Item 1, accounting for 90% of the population at a 95% (or higher) confidence level.

Calculated weight and units with Bayesian Sampling . . .

Item 1	A calculated 417.9 grams of powder from an estimated 2786 packets	Methamphetamine

REMARKS: The Bayesian sampling plan was utilized to analyze Item 1, accounting for 100% of the population at a 95% (or higher) confidence level.

Calculated weight and units with Hypergeometric Sampling . . .

Item 1-1	A calculated 427.1 grams of chunky substance from an estimated 1836 packages	Cocaine
Item 1-2	A calculated 47.4 grams of chunky substance from an estimated 204 packages	Not Analyzed

REMARKS: The Hypergeometric sampling plan was utilized to analyze Item 1, accounting for 90% of the population at a 95% (or higher) confidence level.

REFERENCES

1. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Representative Drug Sampling; 2003.

2. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Sampling of Illicit Drugs for Qualitative Analysis; Second Edition; 2016.

3. Hedayat, A.S. and Zhang, W.G. "Sampling Plans and Related Statistical Inferences of Forensic Drug Study" 1994 Technical Report No. 94-01.

4. Drug Chemistry Training Manual – Section DC-IA-1.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

METHOD: GAS CHROMATOGRAPHY - INFRARED SPECTROMETRY (GC-IR)

Reviewed by:

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Approved by:

_____

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Forensic Sciences Command

INTRODUCTION

When a gas chromatograph is coupled with a Fourier Transform infrared spectrometer, it gives rise to a hyphenated technique known as gas chromatography-infrared spectroscopy, or GC-IR. The GC-IR combines a chromatography separation technique with confirmatory structural information from the infrared spectrometer, allowing for the positive identification of a compound. While the GC-IR would likely not be the first step in the analytical process for routine casework, it could be employed for difficult cases that involve identical-looking GC/MS spectra or co-eluting positional isomers. The instrument can be used with an automated sampler.

The qualitative analysis of unknown substances by GC-IR is accomplished by direct comparison with a standard spectrum for an identification. Comparison of retention time data generated by the GC can be used as a preliminary test. Identifications made using the FT-IR rely on comparisons of peak positions and relative peak intensities between samples and standard spectra. When retention time data is used in conjunction with infrared spectral data, a GC-IR run is still considered a single test for the purposes of meeting the minimum requirement of two tests.

Sample preparation may or may not require extraction. Due to the separating power of the GC, the same vial run on the GC-MS can also be run on the GC-IR. The computer is used to acquire data in a form that can be used by the chemist. It should not alter the fundamental data.

If the analysis cannot occur at the originating laboratory, a representative sample of the substance and/or vial with dried sample is necessary to perform GC-IR analysis. Sample preparation will be performed at the originating laboratory, and then transferred to the closest laboratory with an instrument in service for analysis to be completed. The sample workflow is provided below.

SAFETY CONSIDERATIONS

Standard Laboratory Practices

Use personal protective equipment to cover eyes and skin while working with liquid nitrogen.

PREPARATIONS

The samples should be dissolved in a suitable solvent, e.g., methanol, chloroform, etc.

INSTRUMENTATION

Dani Gas Chromatograph with autosampler

DiscovIR Fourier Transform Infrared Spectrophotometer

Reference: Operator's manual

MINIMUM STANDARDS AND CONTROLS

The following are requirements to meet the minimum standards and controls for gas chromatography – infrared spectroscopy:

I. Documentation

1. A copy of the GC-IR blank report shall be included before each sample run.
2. A copy of the GC-IR sample report, which includes both the chromatogram and the spectrum, will be included in the case file for each sample run. Using the Print Report workbook, at least two peaks will be labeled with the wavenumber of the apex. These peaks should be within the 2000 – 1300 cm^{-1} region, whenever possible. The peaks selected for labeling shall correlate directly to the peaks used for comparison to a reference standard. The print out of the reference standard with peaks labeled will also be included.
3. The gas chromatography conditions, including column type (included in the method name or analytical notes packet - not included by software) and dimensions, injection temperature, injection volume, split ratio, oven temperatures, oven ramps, hold times, gas flow, transfer line temperature, restrictor temperature, and IR parameters including disk temperature, disk speed, data collection time, track pointer, and chamber pressure will be on the report.
4. The item number, analyst's initials, and date will be recorded on the report and all spectra. Item number is optional on the standards and blanks.
5. The worksheet will indicate the result of the IR and any additional spectra will be referenced to the worksheet (excluding standards). Chromatograms and spectra shall be traceable to the GC-IR from which they were generated. Data that is NOT suitable for comparison, but is included in the case file, must be distinguishable from data that is suitable for comparison. This can be accomplished by associating samples with their corresponding spectra and results in the "Notes" panel in LIMS or through annotations on the included spectra.

II. Screening parameters

1. Per the manufacturer, the IR screening parameters are as follows:
   Scan: 1 (real time) as opposed to number of scans
   Resolution: 4 cm^{-1}
   Starting wavenumber: 4000 cm^{-1}
   Ending wavenumber: 700 cm^{-1}

Gain: 1

2. For a general screening run, the injection volume will be 1 microliter (2 microliters, if needed) and the split ratio will be between 5:1 and 20:1.
   a. "Weak" methods that alter the split ratio or injection volume to allow more sample onto the column are acceptable for use after a general screening run has been attempted or determined based on previous GC-MS run.
   b. Methods tailored for the identification of a particular drug (or class of drugs) are exempt from these requirements.

III. Comparisons

1. Any qualitative comparison(s) will be made to in-house standards after the data has been assessed for suitability (see DC-App II).
2. Spectral comparisons
   a. For spectral comparisons (including positive identification, standard reference, and documentation criteria) refer to DC-F-1/III.
3. Retention time comparisons
   a. For retention time comparisons (including positive indications, standard reference, and documentation criteria), refer to DC-D-1/III.

IV. Function Checks/Positive Controls

1. A logbook (or online equivalent) will be kept for the instrument to record maintenance, function (QC) checks, positive controls, standards for comparisons, and column installation date.

   a. A polystyrene standard will be run at least once a month or before operation if not used on a routine basis. The results of this function check are preserved in a data table on the instrument. A passing function check will have at least two polystyrene wavenumbers between 2000 and 1300 cm^{-1} fall within 1 cm^{-1} of the associated NIST reference wavenumbers. A copy of this table (to include the NIST reference numbers and the current polystyrene run) will be maintained in the logbook (or online equivalent).
   b. The GC-IR has two function checks (referred to as QC tests) that should be performed prior to the first sequence of the day:
      1. Align (throughput) test - The purpose of this test is to ensure that the instrument is operating optimally. The results shall be between 4 and 8 volts, with ideal readings being between 5 and 7 volts.
      2. Signal-to-Noise test- This test will help determine if the background is interfering with the sample signal. The

instrument runs this test five times. The average result of these is normally between 0.5 and 1.0 with a maximum upper limit of 1.5. If the signal-to-noise ratio is above 1.5, then the test will be completed again. If the second test fails, then contact vendor for further assistance.

c. Positive controls shall be run at least monthly, or before operation if not used on a routine basis
    1. A program to be used as a general drug screen or unknown run will be checked by running low and high boiling standards. A general screening run shall demonstrate the ability to detect both GHB or GBL and Buprenorphine or Trazodone. **Any change to this method (GC or IR parameters) will require a new positive control to be run.**
    2. The control spectra will be peak-picked and compared to previous peak-picked spectra and maintained in the instrument logbook (or online equivalent). Deviations greater than +/- 1 cm^{-1} of prominent peaks in the 2000-1300 cm^{-1} region from the prior function check will need to be considered. Trending deviations should be investigated and addressed.
    3. The chromatogram must be compared to a previously accepted control or reference material and examined for peak shape, height, and retention time reproducibility.
d. After GC or transfer column installation and/or maintenance (i.e. column clipping), verification of the general screen method will be completed to demonstrate the instrument's ability to detect a range of substances.

PROCEDURE OR ANALYSIS

1. A background spectrum (collected with every run) should be included in the GC-IR report.
    a. A blank is required prior to every sample run on the GC-IR, and the report for each blank will be included in the case file.
    b. Each sample will be run immediately following a blank and will be set to overlay on the same disc location as its associated blank.

SAMPLE SCREENING/UNKNOWN PROGRAM
The following method is an example of a general drug screen. It includes low boilers and high boilers.

Temperatures/Degrees (Celsius)

Injector	250
Disc	-35
Transfer line	280
Restrictor	280
Oven	60 for 0.80 min, ramp to 300°C at 35°C/minute, hold at 300°C for 6.35 min
Column	15M, HP-35, 0.25mm ID, 0.25 µm film
Carrier Gas	Helium
Other Supplies	Liquid Nitrogen

IR parameters (most are hard wired into the instrument [cannot be changed])
Scan: 1 (real time) as opposed to number of scans
Resolution: 4 cm^{-1}
Starting wavenumber: 4000 cm^{-1}
Ending wavenumber: 700 cm^{-1}
Gain: 1

REPORT WORDING

See Appendix I – Report Wording

REFERENCES

1. The Drug Chemistry Training Manual, Section TBD.

2. Abdel-Hay, Karim M., et al. "GC-MS and GC-IRD Studies on the Six-Ring Regioisomeric Dimethoxybenzylpiperazines (DMBPs)." *Drug Testing and Analysis*, vol. 5, no. 7, 2012, pp. 560–572.

3. Shipman, Robert, et al. "Forensic Drug Identification by Gas Chromatography – Infrared Spectroscopy."

4. Bartick, Edward G. "Application of Vibrational Spectroscopy in Criminal Forensic Analysis." *Handbook of Vibrational Spectroscopy,* 2002, pp. 1-11.

5. Dawson, James Zachary. "The Validation of a GC-IR and GC-MS and GC-IR Analysis of Methcathinone Analogs."

6. Drug Identification Procedures Manual, Gas Chromatography-Fourier Transform Infrared Spectroscopy, Georgia Bureau of Investigation, Division of Forensic Sciences

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF SUSPECTED CONTROLLED SUBSTANCES

METHOD: ANALYSIS OF TABLETS AND CAPSULES

PROCEDURE: ANALYSIS OF TABLETS AND CAPSULES

Reviewed by:

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Forensic Sciences Command

INTRODUCTION

Items containing controlled substances in tablets and/or capsules will sometimes be submitted for analysis. One procedure that can be used for the identification of these materials is the Physical Identification procedure. If more than one tablet and/or capsule is to be identified to meet statutory requirements, the required number of tablets and/or capsules must be tested individually. If necessary, the submission may be analyzed using the Bayesian or Hypergeometric sampling plans. The calculated number of units must each be individually tested to meet the Minimum Standards and Controls for analysis.

SAFETY CONSIDERATIONS

No additional safety considerations, just follow the requirements of the selected testing procedure.

PREPARATIONS

Follow the requirements of the selected procedure.

INSTRUMENTATION

Follow the requirements of the selected procedure.

MINIMUM STANDARDS AND CONTROLS

1. When applicable, the case file will reflect the sampling plan used.

2. The population will be determined to be visually homogenous.

3. When applicable, the results of the sampling plan calculations will be included in the case file.

4. Unless using a statistical sampling plan, only the items tested will be identified.

PROCEDURE OR ANALYSIS

1. Follow the "Physical Identification" procedures and should a result be obtained, the analyst will determine if additional testing is required for the charge. If more than one tablet or capsule is needed to meet the statutory limits, the appropriate number of tablets must be tested individually or, if the sample allows, the population may be analyzed using a statistical sampling plan. Hypergeometric Sampling and Bayesian Sampling are examples of acceptable sampling plans.

2. For obviously clandestine dosage units or units for which the "Physical Identification" procedure does not provide a result, analysis will continue using the powder sample method. If more than one tablet or capsule is needed to meet the statutory limits, the appropriate number of tablets must be tested individually or, if the sample allows, the

population may be analyzed using a statistical sampling plan. Hypergeometric Sampling and Bayesian Sampling are examples of acceptable sampling plans.

3. The units to be tested will be randomly selected from the total population. A random number generator may be used.

4. If a negative result is obtained, the statistical sampling plan will be aborted and testing the appropriate number of units necessary for the charge will be done.

REPORT WORDING

The analyst will use a remarks line on the report to indicate the sampling model used.

REFERENCES

1. European Network of Forensic Science Institutes, Drugs Working Group. Guidelines on Representative Drug Sampling; 2003.

2. Coulson, S.A.; Coxon, A.; and Buckleton, J. "How Many Samples from a Drug Seizure Need to be Analyzed?" Jour. For. Sci. 2001; 46(6):1456-1461.

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5. Hedayat, A.S. and Zhang, W.G. "Sampling Plans and Related Statistical Inferences of Forensic Drug Study" 1994 Technical Report No. 94-01.

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8. Clark, A. B. and Clark, C. C. "Sampling of Multi-Unit Drug Exhibits" Jour. For. Sci.. 1990; 35(3): 713-719.

9. Joyce, James, "Bayes' Theorem", *The Stanford Encyclopedia of Philosophy* (Fall 2008 Edition), Edward N. Zalta (ed.), URL = <http://plato.stanford.edu/archives/fall2008/entries/bayes-theorem/>.

10. ASTM E2548-16, Standard Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis, ASTM International, West Conshohocken, PA 2016, www.astm.org

11. From Procedures or Appendices as needed.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF SUSPECTED CONTROLLED SUBSTANCES

METHOD: ANALYSIS OF POWDERED SAMPLES

PROCEDURE: ANALYSIS OF POWDERED SAMPLES

Reviewed by:

_____

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Forensic Sciences Command

INTRODUCTION

Many samples submitted to the laboratory for analysis consist of powders in one form or another, e.g., fine/coarse mixtures, chunky, etc. A characteristic of all of these is the ability to reduce them to homogenous mixtures. However, depending on the situation, it may not be necessary to reduce each sample to a uniform mesh size. The experience of the analyst will determine this as well as the purpose of the analysis (qualitative or quantitative). Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete the identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than fulfill these standards.

After evidence handling procedures have been completed and the mass determined for the sample, a portion can be selected for analysis. The requirement to use less than 50% of the sample in the analysis can be exceeded if the analysis dictates it. However, for small amounts, it may be necessary to save the extracted material used in the analysis.

Once the sample is ready for analysis, the analyst must select the procedures necessary to detect and positively identify or eliminate the presence of any controlled substances. This choice is left up to the analyst as long as the result meets the requirements listed in the general introduction to the analysis of controlled substances. The results of the analysis will be reported as indicated in Appendix I – Report Wording.

SAFETY CONSIDERATIONS

Follow the requirements of selected procedure.

PREPARATIONS

Follow the requirements of each procedure. It may be necessary to extract samples prior to analysis.

INSTRUMENTATION

Follow the requirements of selected procedure.

MINIMUM STANDARDS AND CONTROLS

Follow the requirements of selected procedure.

PROCEDURE OR ANALYSIS

Follow the requirements of selected procedure.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual.
2. From Procedures or Appendices as needed.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF SUSPECTED CONTROLLED SUBSTANCES

METHOD: ANALYSIS OF LIQUIDS, PAPER, NON-CANNABIS PLANT MATERIAL, RESIDUES, AND FOOD PRODUCTS

PROCEDURE: ANALYSIS OF LIQUIDS, PAPER, NON-CANNABIS PLANT MATERIAL, RESIDUES, AND FOOD PRODUCTS

Reviewed by:

_____

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Approved by:

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INTRODUCTION

Liquids, Non-Cannabis Plant Material, Samples on Paper, Residues, or Food Products are some general types of evidence submissions. Each of these has particular handling or extraction procedures, but all are analyzed in a similar manner after isolation of the controlled substance from the initial matrix. The following are some general extraction procedures for the analysis of these types of samples.

Liquids:	Sometimes can be extracted using chloroform or similar organic solvent. The addition of dilute acid or base solutions to the initial aqueous matrix can assist both the extraction and help in diluting the sample. Other procedures can be found in the Appendix III under Extractions and Separations.
Paper:	This usually consists of LSD, an NBOMe compound, or a synthetic Cannabinoid applied to various types of paper. The sample can be directly extracted with an organic solvent and sonication or extracted with ammonium hydroxide solution and chloroform. See Appendix III for other extractions. Generally, the paper being analyzed should be weighed in its entirety and reported appropriately. There may also be times when the paper can be considered a residue. When that occurs, the analyst will analyze and report the paper as a residue.
Residues:	These can be analyzed with various wash techniques e.g. methanol, and the volume reduced prior to analysis. In order for residues to be analyzed, a request from the State's Attorneys Office must be submitted and approved by the Lab Director or designee.
Non-Cannabis Plant Material:	These may require some blending, grinding, or sample reduction of the plant tissue prior to extraction. Once the matrix is broken down, organic solvents and extractions can sometimes help remove the controlled substance.
Food Products:	Food products vary from candies spotted with suspected LSD or NBOMe, gummy candies containing benzodiazepines, to commercially prepared foods containing THC. Care should be taken to minimize the impact of the sample matrix on instrumentation through careful preparation and extractions.

SAFETY CONSIDERATIONS

Follow the requirements of selected procedure.

PREPARATIONS

This will depend on the matrix.

INSTRUMENTATION

This will depend on the matrix.

MINIMUM STANDARDS AND CONTROLS

Follow the requirements of selected procedure.

PROCEDURE OR ANALYSIS

Follow the requirements of selected procedure.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual.
2. From Procedures or Appendices as needed.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF A CONTROLLED SUBSTANCE

METHOD: QUANTITATION

PROCEDURE: CENTRALIZED QUANTITATION

Reviewed by:

_____

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INTRODUCTION

The quantitation of scheduled substances will only be performed at a centralized laboratory location. This section covers the protocol for sampling a case for quantitation. The procedure used for quantitation of a scheduled substance is at the discretion of the chemist as long as minimum standards and control criteria are met.

A representative sample of the entire submitted substance is necessary to perform quantitation. Sample preparation will be performed at the originating laboratory, and then transferred to the centralized laboratory for quantitation.

SAFETY CONSIDERATIONS

Standard Laboratory Practices for Use of Solvents. Refer to current DOT and Postal requirements when shipping caustic liquid samples.

PREPARATIONS

None Required.

INSTRUMENTATION

Balances

GC-FID with or without autosampler

GC-MS with or without autosampler

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

The following are general minimum standards and controls for quantitation:

1. Quantitation will be performed using proven methods which are accepted by the scientific community.

2. Quantitation will be performed only upon approval by laboratory management of both the submitting laboratory and the centralized quantitation laboratory. For the Forensic Science Center at Chicago, this is the Section Chief or appropriate designee. Only one single item will be quantitated.

3. Calculations will be performed using only scientifically approved methods. All quantitation results will be reported to two significant figures (i.e. a sample of 56.75% will be reported as 56% and a sample of 5.67% will be reported as 5.6%)

4. Instrumental linearity will be established.

5. Each centralized quantitation Drug Chemistry Section will incorporate in-house quantitative procedures, including uncertainty measurement and proper use and tracking of quantitation reference materials, into the Facility Operations Manual. These procedures will be available for inspection by the quality review coordinator(s) and other inspection teams.

6. Volumetric glassware used for the preparation of quantitation samples will be checked through the appropriate creation of a calibration curve and analysis of positive control samples. In addition, visual checks of the volumetric glassware will be done prior to each use and any damaged volumetrics will be discarded.

7. A control chart must be prepared and measurement uncertainty determined (as outlined in Appendix V) for a quantitation procedure and drug before such analysis can be performed in casework.

8. A control sample must be analyzed with each case (as outlined in Appendix V). The control should come from the same stock that was used to create the control chart. If that control sample is unavailable, a new sample must be prepared and a new control chart created.

9. The control sample must fall within 2 standard deviations (2 σ) on the control chart for the analysis to be valid.

PROCEDURE OR ANALYSIS

1. All quantitations will have laboratory management / supervisor approval. Qualitative analysis must be completed prior to consideration for quantitation. If a request for quantitation as a supplemental report is granted, the case will be resubmitted to the originating laboratory for sample preparation prior to transferring to the centralized quantitation laboratory.

2. Collect a representative sample. The method used for sample collection will be recorded on the worksheet; e.g. blender, crushing and sifting, crushing mortar and pestle, etc. An acceptable range of the subsample is no less than 1 to 5 grams.

   A. For powder samples, the entire sample will be crushed and the subsample (target amount is 4 grams) will be made homogenous.

   B. Liquid samples should be placed into disposable containers. If there are two separate layers then each layer will be pipetted separately into its own disposable container.

3. Assign the subsample an identification subitem number in LIMS from the original case item number. Record the mass of the subitem number in LIMS. The mass of the subitem will not be listed on the laboratory report.

4. Complete the Quantitation Sample Collection Form using the form located in DC-IIA-1. The form will be sent to the centralized laboratory and placed in the case file.

5. Notify the receiving laboratory/person of the quantitation.

6. Relocate the subitems in LIMS.

7. Prepare and transfer the sample using necessary precautions to prevent contamination and to maintain integrity of evidence. If needed, also be sure to meet US Postal and DOT regulations.

8. Analysis of the subitem at the centralized location will be performed using an approved quantitation procedure.

9. The subitem will be tracked through LIMS. If the centralized quantitation site is the laboratory from which the case originated, return of the quantitation sample with the original submission is an option.

10. The centralized laboratory will issue a report using report wording guidelines found in DC-Appendix I. If the report is supplemental, the report will reflect the original report date and name of the original analyst. An optional evidence disposition statement may be added stating how evidence will be returned.

SHIPPING PRECAUTIONS

The following precautions will be observed when shipping samples:

1. All cases will be transferred using current DOT and postal requirements.

2. All powder samples must be placed in a heat-sealed plastic bag. All liquid samples must be placed in airtight containers (i.e. paint cans with friction lids, glass jars with tight fitting screw lids) before placing in outer packaging.

3. Always pack liquid products in sufficient cushioning and absorbent material to absorb any breakage and, as necessary, to conform to DOT regulations for shipping of hazardous materials.

4. No pressurized canisters will be shipped.

5. Consult your supervisor if you have any packaging concerns.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual – Section DC-IA-7.

2. Skoog, D.; West, D.; Holler, F. J. "Fundamentals of Analytical Chemistry, 5th ed., Saunders College Publishing, New York, NY, 1988.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF A CONTROLLED SUBSTANCE

METHOD: QUANTITATION

PROCEDURE: CENTRALIZED QUANTITATION

Reviewed by:

_____

Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

The quantitation of scheduled substances will only be performed at a centralized laboratory location. This section covers the protocol for sampling a case for quantitation. The procedure used for quantitation of a scheduled substance is at the discretion of the chemist as long as minimum standards and control criteria are met.

A representative sample of the entire submitted substance is necessary to perform quantitation. Sample preparation will be performed at the originating laboratory, and then transferred to the centralized laboratory for quantitation.

SAFETY CONSIDERATIONS

Standard Laboratory Practices for Use of Solvents. Refer to current DOT and Postal requirements when shipping caustic liquid samples.

PREPARATIONS

None Required.

INSTRUMENTATION

Balances

GC-FID with or without autosampler

GC-MS with or without autosampler

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

The following are general minimum standards and controls for quantitation:

1. Quantitation will be performed using proven methods which are accepted by the scientific community.

2. Quantitation will be performed only upon approval by laboratory management of both the submitting laboratory and the centralized quantitation laboratory. For the Forensic Science Center at Chicago, this is the Section Chief or appropriate designee. Only one single item will be quantitated.

3. Calculations will be performed using only scientifically approved methods. All quantitation results will be reported to two significant figures (i.e. a sample of 56.75% will be reported as 56% and a sample of 5.67% will be reported as 5.6%)

4. Instrumental linearity will be established.

5. Each centralized quantitation Drug Chemistry Section will incorporate in-house quantitative procedures, including uncertainty measurement and proper use and tracking of quantitation reference materials, into the Facility Operations Manual. These procedures will be available for inspection by the quality review coordinator(s) and other inspection teams.

6. Volumetric glassware used for the preparation of quantitation samples will be checked through the appropriate creation of a calibration curve and analysis of positive control samples. In addition, visual checks of the volumetric glassware will be done prior to each use and any damaged volumetrics will be discarded.

7. A control chart must be prepared and measurement uncertainty determined (as outlined in Appendix V) for a quantitation procedure and drug before such analysis can be performed in casework.

8. A control sample must be analyzed with each case (as outlined in Appendix V). The control should come from the same stock that was used to create the control chart. If that control sample is unavailable, a new sample must be prepared and a new control chart created.

9. The control sample must fall within 2 standard deviations (2 σ) on the control chart for the analysis to be valid.

PROCEDURE OR ANALYSIS

1. All quantitations will have laboratory management / supervisor approval. Qualitative analysis must be completed prior to consideration for quantitation. If a request for quantitation as a supplemental report is granted, the case will be resubmitted to the originating laboratory for sample preparation prior to transferring to the centralized quantitation laboratory.

2. Collect a representative sample. The method used for sample collection will be recorded on the worksheet; e.g. blender, crushing and sifting, crushing mortar and pestle, etc. An acceptable range of the subsample is no less than 1 to 5 grams.

   A. For powder samples, the entire sample will be crushed and the subsample (target amount is 4 grams) will be made homogenous.

   B. Liquid samples should be placed into disposable containers. If there are two separate layers then each layer will be pipetted separately into its own disposable container.

3. Assign the subsample an identification subitem number in LIMS from the original case item number. Record the mass of the subitem number in LIMS. The mass of the subitem will not be listed on the laboratory report.

4. Complete the Quantitation Sample Collection Form using the form located in DC-IIA-1. The form will be sent to the centralized laboratory and placed in the case file.

5. Notify the receiving laboratory/person of the quantitation.

6. Relocate the subitems in LIMS.

7. Prepare and transfer the sample using necessary precautions to prevent contamination and to maintain integrity of evidence. If needed, also be sure to meet US Postal and DOT regulations.

8. Analysis of the subitem at the centralized location will be performed using an approved quantitation procedure.

9. The subitem will be tracked through LIMS. If the centralized quantitation site is the laboratory from which the case originated, return of the quantitation sample with the original submission is an option.

10. The centralized laboratory will issue a report using report wording guidelines found in DC-Appendix I. If the report is supplemental, the report will reflect the original report date and name of the original analyst. An optional evidence disposition statement may be added stating how evidence will be returned.

SHIPPING PRECAUTIONS

The following precautions will be observed when shipping samples:

1. All cases will be transferred using current DOT and postal requirements.

2. All powder samples must be placed in a heat-sealed plastic bag. All liquid samples must be placed in airtight containers (i.e. paint cans with friction lids, glass jars with tight fitting screw lids) before placing in outer packaging.

3. Always pack liquid products in sufficient cushioning and absorbent material to absorb any breakage and, as necessary, to conform to DOT regulations for shipping of hazardous materials.

4. No pressurized canisters will be shipped.

5. Consult your supervisor if you have any packaging concerns.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual – Section DC-IA-7.

2. Skoog, D.; West, D.; Holler, F. J. "Fundamentals of Analytical Chemistry, 5th ed., Saunders College Publishing, New York, NY, 1988.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF A CONTROLLED SUBSTANCE

METHOD: QUANTITATION

PROCEDURE: QUANTITATION BY GAS CHROMATOGRAPHY

Reviewed by:

_____

Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

For most samples, gas chromatography (GC) is the method of choice, since it provides both separation and quantitation of mixtures at one time. There are two methods for quantitation by GC, the external and internal standard methods.

The columns, conditions for the gas chromatograph, solvents and internal standards should be chosen based on the sample being tested. These factors are left to the discretion of the chemist.

Solvents should be chosen that will insure total dissolution of the substance being quantitated. In many cases chloroform is preferred over methanol because sugars are generally insoluble in the chloroform. A mixture of chloroform and methanol can also be used.

To insure that the entire sample injected into the GC is being measured, care must be taken to insure that sodium and sulfate salts are dissociated fully. This problem can be eliminated by adding either a drop of acetic acid or sodium hydroxide to the solution before injection onto the gas chromatograph.

Multiple injections of the standard should be made to insure reproducibility of the system and method. A concentration curve of standards ranging from 0.5 mg/mL to 1.5 mg/mL can be done to test the linearity.

SAFETY CONSIDERATIONS

Standard Laboratory Practices including the use of solvents.

PREPARATIONS

Preparations for Internal Standard Procedure

1. Prepare the internal standard solution.

   The internal standard solution should be at a concentration of approximately 1 mg/mL. A large enough quantity should be made in order to dilute all samples (the standard and the unknown solutions). The internal standard should elute at an appropriate time, have no interferences, and provide a reproducible peak for quantitation. Examples of hydrocarbons which can be used as internal standards are tetracosane, docosane, nonadecane. Other substances could be used - for example propiophenone (P-1-P) can be used for quantitating methamphetamine or dipentyl phthalate for cocaine.

2. Prepare the standard solution.

   A primary standard should be used. An exact amount is weighed on an analytical balance, recorded, and diluted with internal standard by using a repipetter or volumetric flask. The concentration of the standard should be approximately 1 mg/mL.

3. Prepare the unknown solution.

   The salt form of the unknown should be determined by the analyst. If it is a different salt than the standard being used, adjustments for the differences in molecular weights must be performed.

   The sample must be made homogenous before any analysis is performed. The unknown solution is prepared in the same manner as the standard solution. The concentration of the substance being quantitated should be approximately 1 mg/mL.

   To determine the amount of unknown sample to use, its purity is approximated. To obtain a 1 mg/mL sample, the following table can be used:

Estimated Purity	Amount sample (for 1 mL internal standard solution
10%	10.0 mg
25%	4.0 mg
50%	2.0 mg
75%	1.3 mg
100%	1.0 mg

Preparations for External Standard Procedure

The unknown and standard solutions are made as in the internal standard method except an internal standard solution is not used. The unknown and standard are each diluted with solvent only. Both resulting solutions are injected on the gas chromatograph and the areas of the resulting peaks are obtained.

INSTRUMENTATION

Balance

GC with or without autosampler

MINIMUM STANDARDS AND CONTROLS

Refer to Gas Chromatography Procedure for instrument minimum standards and controls.

The following are general minimum standards and controls for quantitation:

1. Quantitation will be performed using proven methods.

   The GC Program used for quantitation will depend upon the substances being quantitated and vary with the column diameter and the conditions. It is impractical to list all of the possible combinations.

   An example of a program for the quantitation of cocaine on an Agilent 6890 is given below:

Instrument:	Agilent 6890
Detector:	FID - 280°
Injector:	250°
Column:	HP-1, 15 meter, 0.53 mm diameter, 2.65 micron coating
Other Conditions:	210° isothermal; split 10:1; flow 11.7 mLs/min Helium
Length of Run:	Approximately 3 minutes

2. Quantitation will be performed only upon approval by laboratory management of both the submitting laboratory and the centralized quantitation laboratory. For the Forensic Science Center at Chicago, this is the Section Chief or appropriate designee. Only one single item will be quantitated.

3. Calculations will be performed using only scientifically approved methods. All quantitation results will be reported to two significant figures (i.e. a sample of 56.75% will be reported as 56% and a sample of 5.67% will be reported as 5.6%)

4. Instrumental linearity will be established.

5. Each centralized quantitation Drug Chemistry Section will incorporate in-house quantitative procedures, including uncertainty measurement and proper use and tracking of quantitation reference materials, into the Facility Operations Manual. These procedures will be available for inspection by the quality review coordinator(s) and other inspection teams. Ultimate responsibility for accuracy in quantitation will rest with the analyst and the laboratory director/section chief.

6. Volumetric glassware used for the preparation of quantitation samples will be checked through the appropriate creation of a calibration curve and analysis of positive control

samples. In addition, visual checks of the volumetric glassware will be done prior to each use and any damaged volumetrics will be discarded.

7. A control chart must be prepared and measurement uncertainty determined (as outlined in Appendix V) for a quantitation procedure and drug before such analysis can be performed in casework.

8. A control sample must be analyzed with each case (as outlined in Appendix V). The control should come from the same stock that was used to create the control chart. If that control sample is unavailable, a new sample must be prepared and a new control chart created.

9. The control sample must fall within 2 standard deviations (2 σ) on the control chart for the analysis to be valid.

PROCEDURE OR ANALYSIS

Procedure for Internal Standard Method

An internal standard is added to the unknown, control, and the standard solutions. Each solution is injected on the gas chromatograph and the areas of the resulting peaks are obtained. These peak areas and the weights of the unknown sample and standard are used to calculate the purity of the sample. Calculations are performed as described below.

Multiple injections of the standard, control and unknown are made. Each solution should be run at least twice. The integration of the areas of the eluted peaks is recorded.

Calculation

The responses of the standard and the unknown are normalized to the internal standard using the peak areas from the GC chromatogram.

Rstd = Area standard/Area of its internal standard

Runk = Area unknown/Area of its internal standard

Multiple runs are calculated separately and then averaged together. These average R values are inserted in the following formula to determine the concentration of the unknown:

$$\left[\frac{R_{unk}}{R_{std}}\right]\left[\frac{\text{conc std}}{\text{conc unk}}\right]\left[X\right] =$$

The symbol X provides the units for the quantitation. If X is 100%, the calculation is percent as the standard salt. If X is the average weight of a tablet, the calculation is mg of the standard salt per tablet. If X is 1, the calculation is mg/mL.

If the salt form of the unknown and the standard are different, adjustments for the differences in molecular weights must be performed. The final result is multiplied by the molecular weight of the salt form of the unknown and divided by the molecular weight of the standard salt form.

Rather than weighing a single standard at 1 mg/mL, a range of standards can be prepared and injected to create a standard curve. The concentrations of the standard samples and the areas of the eluted peaks are used to create a calibration table. This table is stored on the computer. The unknown is then prepared and injected. The areas of the eluted peaks are compared back to the table to determine the amount of unknown in the sample. No manual calculations are necessary.

Procedure for External Standard

This procedure is similar to the internal standard method except that no internal standard is added to the solutions. This method is reliable if automatic injection techniques are used to insure reproducibility. The calculations assume that the injection size for both the unknown and the standard are the same. If not, the calculations must correct the responses for this difference.

Calculation

Multiple runs are performed and the resulting peak areas averaged. The following formula is used to determine the concentration of the unknown:

$$\left[\frac{\text{Area unk}}{\text{Area std}}\right]\left[\frac{\text{conc std}}{\text{conc unk}}\right]\left[X\right] =$$

The symbol X is defined in the internal standard method. If the injection sizes of the standard and unknown are not equal, the area of each must be normalized by dividing by its injection size.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual – Section DC-IA-7.

2. Grob, R. L. Modern Practice of Gas Chromatography, 2nd ed., John Wiley and Sons, New York, NY, 1985, pp. 390-397, 409-412.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF A CONTROLLED SUBSTANCE

METHOD: QUANTITATION

PROCEDURE: QUANTITATION BY GC-MS SELECTED ION MONITORING (SIM)

Reviewed by:

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Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

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Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

The most common method of scanning with GC-MS is to scan a fairly wide range of mass to charge in order to obtain a complete mass spectrum for identification purposes (see Mass Spectrometry method). With selected ion monitoring (SIM) the mass spectrometer is directed to scan only a preselected range or number of masses. Because of this, the mass spectrometer spends its signal collection time only on masses of interest. One can take advantage of this increased signal to noise ratio to enhance the sensitivity of the instrument. In addition to increased sensitivity, the SIM Method also is used to quantitate samples. This is the purpose of this method. An example of this type of analysis for methamphetamine quantitation is presented below. SIM quantitation can be used for other drugs, e.g., cocaine, heroin, etc., with the appropriate internal standard.

SAFETY CONSIDERATIONS

Standard Laboratory Practices including the use of solvents.

PREPARATIONS

Reagents

Propiophenone (P-1-P)
Chloroform
Methamphetamine HCl (Crystalline)
Sodium Hydroxide

0.5 N NaOH solution – 20 grams of Sodium Hydroxide pellets or flakes are dissolved with 1 liter of distilled water. This will generate some heat and should be done with stirring.

Preparations for Internal Standard Procedure

1. Prepare the internal standard solution.

An internal standard solution of propiophenone (P-1-P) is prepared by adding 1.3 mL of P-1-P and diluting to 1 liter with chloroform. This is the base for all solutions.

2. Prepare the standard solution.

A standard solution of methamphetamine HCl is prepared by adding 25 mg of primary standard to 25 mL of previously prepared internal standard solution. Any amount can be prepared as long as the 1 mg/mL ratio is kept.

3. Prepare the unknown solution.

The salt form of the unknown should be determined by the analyst. If it is a different salt than the standard being used, adjustments for the differences in molecular weights must be performed.

The sample must be made homogenous before any analysis is performed. The unknown solution is prepared in the same manner as the standard solution. The concentration of the substance being quantitated should be approximately 1 mg/mL.

To determine the amount of unknown sample to use, its purity is approximated. To obtain a 1 mg/mL sample, the following table can be used:

Estimated Purity	Amount sample (for 1 mL internal standard solution
10%	10.0 mg
25%	4.0 mg
50%	2.0 mg
75%	1.3 mg
100%	1.0 mg

INSTRUMENTATION

GC-MS in SIM mode.

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

Refer to Mass Spectrometry Procedure for instrument minimum standards and controls.

The minimum general standards and controls for quantitation are the following:

1. Quantitation will be performed using proven methods.

2. Quantitation will be performed only upon approval by laboratory management of both the submitting laboratory and the centralized quantitation laboratory. For the Forensic Science Center at Chicago, this is the Section Chief or appropriate designee. Only one single item will be quantitated.

3. Calculations will be performed using only scientifically approved methods. All quantitation results will be reported to two significant figures (i.e. a sample of 56.75% will be reported as 56% and a sample of 5.67% will be reported as 5.6%)

4. Instrumental linearity will be established.

5. Each centralized quantitation Drug Chemistry Section will incorporate in-house quantitative procedures, including uncertainty measurement and proper use and tracking of quantitation reference materials, into the Facility Operations Manual. These procedures will be available for inspection by the quality review coordinator(s) and other inspection teams. Ultimate responsibility for accuracy in quantitation will rest with the analyst and the laboratory director/section chief.

6. Volumetric glassware used for the preparation of quantitation samples will be checked through the appropriate creation of a calibration curve and analysis of positive control samples. In addition, visual checks of the volumetric glassware will be done prior to each use and any damaged volumetrics will be discarded.

7. A control chart must be prepared and measurement uncertainty determined for a quantitation procedure and specific drug before such analysis can be performed in casework.

8. A control sample must be analyzed with each case. The control should come from the same stock that was used to create the control chart. If that control sample is unavailable, a new sample must be prepared and a new control chart created.

9. The control sample must fall within 2 standard deviations (2 σ) on the control chart for the analysis to be valid.

PROCEDURE OR ANALYSIS

Once the solutions (standard, sample, and control) have been prepared they are extracted in the following manner:

1. Approximately 1 mL of solution is placed in a culture tube (about 1/3 of a 10 x 75 mm size) and an equal amount of 0.5 N NaOH is added and mixed together.

2. The tube is centrifuged and the chloroform layer removed. This chloroform is centrifuged twice more to remove water.

3. The chloroform solution is transferred to an autosampler vial.

The vial is placed on mass spectrometer for analysis. The following is an example of the conditions for an Agilent GC-MSD.

GC

Column:	HP-1 capillary, 12m, 0.2 mm I.D., 0.33 micrometer
Carrier Gas:	Helium
Inlet:	230°C
Detector:	280°C
Isothermal temperature:	100°C

MSD

Ions scanned:	58, 77, 91, 105, 134, 148
Dwell time:	100
Integration:	T = 16

Inject the standard methamphetamine solution and integrate the areas of the peaks. Choose the one ion in P-1-P and the one ion in methamphetamine that are closest in area (response). This is usually the 105 ion for P-1-P and the 58 ion for methamphetamine. Make multiple injections of all solutions (the standard methamphetamine, the control methamphetamine, and the sample being tested) and average the integrated areas of the selected ions for each.

Calculation

The integrated areas of mass 105 for P-1-P and mass 58 for methamphetamine are ratioed:

$$R = \frac{\text{area 58 peak}}{\text{area 105 peak}}$$

The concentration of methamphetamine in the sample is obtained using this equation:

$$\%\ \text{Methamphetamine HCl} = \frac{\text{average R unknown}}{\text{average R std}} \times \frac{\text{mg/mL std}}{\text{mg/mL unk}} \times 100$$

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual – Section DC-IA-9.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF A CONTROLLED SUBSTANCE

METHOD: DETERMINATION OF OPTICAL ISOMERS

PROCEDURE: POLARIMETRY

Reviewed by:

_____

Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

For some substances which exist as enantiomers, only one of the pair is controlled e.g., dextropropoxyphene. The polarimeter is used to determine the direction of rotation of plane polarized light, which defines the enantiomer present. It is the analyst's responsibility to ensure the rotation is due to the substance being tested. Many substances, including sugars, are optically active compounds.

SAFETY CONSIDERATIONS

Standard Laboratory Practices for handling solvents.

PREPARATIONS

The sample is dissolved in chloroform, methanol, or other suitable solvents.

Note: Problems with optical activity have been encountered while using methanol as a solvent. Sugars, methamphetamine, and related compounds can be affected.

INSTRUMENTATION

Polarimeter
Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

The following are the minimum standards and controls for the use of the polarimeter:

1. The worksheet will reflect the direction of rotation.

2. The light source (sodium or mercury) and wavelength will be recorded on the worksheet/work notes.

3. The solvent will be recorded if it is other than methanol.

4. A logbook (or online equivalent) will be kept for the instrument to record maintenance and function checks. Either the sodium or mercury lamp as well as the optical rotation should be checked when the instrument is used.

PROCEDURE OR ANALYSIS

Note: The instrument should be tested to ensure proper results. An optical rotation test must be performed prior to running an unknown. To ensure stabilization, the power and lamps should be turned on for one hour prior to taking a reading.

1. Zero the instrument with a solvent blank.

2. The unknown is dissolved in a suitable solvent and filtered if necessary. Place the solution in a polarimeter cell. The cell is placed in the polarimeter and allowed to stabilize.

3. The direction of rotation is noted. Several different wavelengths can be used if the degree of rotation is small.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Drug Chemistry Training Manual – Section DC-IA-15.

2. Willard, L. Instrumental Method of Analysis, 6th ed., 1981, Wadsworth Publishing: Belmont, CA, pp. 412-427.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF SUSPECTED CANNABIS SAMPLES

METHOD: THE IDENTIFICATION OF CANNABIS

PROCEDURE: BOTANICAL OBSERVATION OF LEAFY PLANT MATERIAL

Reviewed by:

_____

Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

INTRODUCTION

Cannabis is controlled in the state of Illinois under the Cannabis Control Act (CCA), Chapter 720, Act 550 and the Cannabis Regulation and Tax Act (CRTA), Chapter 410, Act 705 of the Illinois Compiled Statutes. This is presented as a reference to be referred to for weight limits, penalties, and other specific requirements of the law.

The term Cannabis, when it appears on a report, has the same definition as that stated in the Cannabis Control Act.

Macroscopic and microscopic examination of plant material can be incorporated into an analytical scheme to positively identify Cannabis. For identification criteria and other guidelines for Cannabis analysis see Appendix IV-B, the Cannabis Minimum Standards and Controls.

Other Related Procedures: Duquenois-Levine Test, Thin-Layer Chromatography, Gas Chromatography, Gas Chromatography-Mass Spectrometry.

SAFETY CONSIDERATIONS

Warning: A fungi, Aspergillas fumigates, may be found on decaying plant material. Spores are released when the plant material is removed from its packaging. Breathing these spores may result in aspergillosis, which affects the pulmonary system in different ways. Wearing a dust mask and/or working in a ventilation hood is advisable.

PREPARATIONS

Not Required.

INSTRUMENTATION

Stereoscope

Reference: Operator's Manual

MINIMUM STANDARDS AND CONTROLS

1. See Chemistry Minimums Standards and Controls: Administrative Policies for Cannabis Analysis (Appendix IV-B) for general minimum standards for Cannabis identification.

2. For both macroscopic and microscopic observations, positive or negative results will be recorded on the worksheet and must include a description of the observed botanical details.

PROCEDURE OR ANALYSIS

If possible, the plant material should be viewed in its entirety, with and without magnification, to determine uniformity.

Macroscopic Examination

1. Gross morphological characteristics that may be observed include the palmate arrangement of the leaflets, the pinnate leaf venation, the serrated edges of the leaflet, the buds (with or without seeds) and, if present, fluted stems and stalks. Stems, a support structure for another part of the plant such as a leaf or flower may also have hairs on the surface.

2. Due to the compressed or processed nature of many samples, some of these characteristics may not be discernible.

3. Plant material shall be considered to have positive macroscopic examination results consistent with Cannabis when at least one of the above characteristics is observed.

    a. Positive or negative results will be recorded on the worksheet and must include a description of the observed macroscopic details.

Microscopic Examination

1. The plant material is observed under a stereoscope with a magnification strong enough to determine the necessary characteristics.

2. The microscopic test is positive for the presence of Cannabis when cystolithic hairs and covering hairs are present on opposing surfaces of the same leaf.

    a. Positive or negative results will be recorded on the worksheet and must include a description of the observed microscopic details.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Arizona Department of Safety – Information Bulletin, 85-01, 1985.

2. U. S. Internal Revenue Service, Description of the Plant, IRS 973004-51-3, Washington, D.C., 1951: pp. 1-10.

3. Thornton, J. and Nakamura G. “Journal of the Forensic Science Society”, Vol. 12, No. 3, 1972, pp. 461-519.

4. Stern, William, author, Joyce, G. R.B., and Curry, S. H. eds., The Botany and Chemistry of Cannabis, 1970, pp. 1-10.

5. Quimby, Maynard W., et al, “Mississippi Grown Marijuana – Cannabis Sativa Cultivation and Observed Morphological Variations”, Econ. Bot., Vol. 27, 1973, pp. 117-127.

6. Clarke, R., The Botany and Ecology of Cannabis, Podds Press, Ben Lomond, CA, 1977, pp. 1-10, 150-153, and 163-168.

7. Drug Chemistry Training Manual – Section DC-IA-2.

8. Illinois Criminal Law and Procedures. Current Edition: Section 720, Act 550, West Publishing Corporation, St. Paul, MN

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

PROTOCOL: ANALYSIS OF SUSPECTED CANNABIS SAMPLES

METHOD: THE IDENTIFICATION OF CANNABIS

PROCEDURE: THE USE OF THE DUQUENOIS-LEVINE TEST FOR THE IDENTIFICATION OF CANNABIS

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INTRODUCTION

The Duquenois-Levine test can be used for the identification of Cannabinoids. To identify samples as Cannabis, two positive tests are acceptable if one of the tests provides structural confirmation (e.g. GC-MS). A positive Duquenois-Levine test is one testing option for the identification of Cannabis and Cannabinoids.

SAFETY CONSIDERATIONS

Standard Laboratory Practices.

PREPARATIONS

Vanillin. Crystalline product (99%)
95% Ethanol
Acetaldehyde
Concentrated Hydrochloric Acid
Chloroform

Note: Selected Chloroform must not contain Amylene as a preservative. Use only Ethanol Stabilized Chloroform.

Duquenois Reagent: One gram of vanillin is added to 50 mL of absolute or 95% ethanol. To this solution, 0.6 mL of cold acetaldehyde is added and the solution mixed together. The 0.6 mL of acetaldehyde is not critical and may be approximated by 12 drops.

INSTRUMENTATION

No Instrumentation Required.

MINIMUM STANDARDS AND CONTROLS

1. See Chemistry Minimums Standards and Controls: Administrative Policies for Cannabis Analysis (Appendix IV-B) for general minimum standards for Cannabis identification.

2. The prepared Duquenois Reagent will be labeled with the preparer's identity, and date made and expiration date.

3. The Duquenois Reagent will be tested when prepared and as indicated in QM-14, using primary or secondary standards. A logbook (or online equivalent) will be kept of the testing to include the identity of the preparer, the date the reagent was

authenticated (or re-authenticated), and the expiration date of the reagent. Any manufactured reagents will be tested when opened and as indicated in QM-14; if no manufacturer's expiration date is given, it will be assigned a one year date and treated in the same manner as in-house reagents.

4. The observed color change and transfer will be documented on the worksheet to indicate results (e.g. purple / purple).

PROCEDURE OR ANALYSIS

The Duquenois-Levine test is conducted in three steps:

Step 1: Extract the sample using petroleum ether. Other solvents, such as methanol, hexane, chloroform, or methylene chloride may be used to extract the sample without affecting the results of the test.

Step 2: Dry the extract, add Duquenois Reagent followed by concentrated Hydrochloric Acid in approximately equal amounts. Wait up to 2 minutes and note the color, if any, that develops.

Step 3: Add chloroform. Note the color, if any, that transfers to the chloroform layer. *See note on Chloroform in Preparations section.

Though the colors for this test are somewhat subjective, a positive result ranges from a blue to purple for the top layer coupled with a transfer of a purple to blue color to the bottom chloroform layer. The colors formed are due to the concentrations of the various Cannabinoids present and can vary with the age and condition of the sample.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

1. Duquenois, P., Moustapha, H. N., "Journal of the Egyptian Medical Association', Vol. 21, pp. 224-227, 1938 (French).

2. Official Methods of the AOAC, 14th ed., AOAC, Washington D. C., 1984, p. 774.

3. Butler, W., "Journal of the AOAC", Vol. 45, No. 3, 1962, pp. 597-600.

4. Thornton, J. and Nakamura G. "Journal of the Forensic Science Society", 1972, Vol. 12, No. 3, pp. 461-519.

5. Drug Chemistry Training Manual – Section DC-IA-2.

6. Chemical Companies:

Sigma-Aldrich Chemical Company
P O Box 14508
St. Louis, MO 63178-4508
1-800-325-3010

Fisher Scientific
300 Industry Drive
Pittsburgh, PA 15275
1-800-766-7000

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APPENDIX I: REPORT WORDING

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APPENDIX I

REPORT WORDING

The report represents a summary of analytical findings. It does not include all information contained in the notes. The report includes the following:

1. The weight of the substance is reported to one tenth of a gram, unless more crucial figures are needed for charge determination (with the exception of some bulk balances). If the mass is less than 0.1 gram, it shall be reported as either less than 0.1 gram or a residue. When a Sampling Plan is utilized and the weight is determined, a calculated weight may be reported.

2. The controlled substance identified is reported as it is found in the Controlled Substances Act. Salt forms are listed only when necessary. The most common example of the appropriate use of a salt form is when quantitation is reported or when reporting cocaine base. For substances that are controlled via their chemical classification, the findings must include the name of the substance and its classification in the law.

3. Additional information may be included if necessary. The following is a summary of the criteria and recommended report wording for various drug chemistry results.

4. There are additional report wordings examples in the Sampling Section of the Procedures Manual (H-1, H-1A, H-1B and H-1C).

CRITERIA	RECOMMENDED REPORT FORMAT
1. Positive Identification	
a. Drugs	Substance name (Abbreviation)
	Substance name (Abbreviation), a [classification from the law] compound
b. Plant material	Cannabis (specific cannabinoid)
	Chemical name (Abbreviation)
c. Quantitation XXX* (compound)	XXX* (% calculated as Y†)

2. No Identification

a. Suspected Drugs	No Scheduled Substance Found
	No Scheduled Substance Indicated
	Physically resemble pharmaceutical preparations which do not contain a scheduled substance
	Physically resembles a pharmaceutical preparation which does not contain a scheduled substance
	Physically resembles a pharmaceutical preparation containing XXX*; no further analysis
b. Suspected Cannabis	No Cannabis Found
	Not Cannabis
c. Drugs and Cannabis	No other controlled substances found
	No other scheduled substance found
	Testing did not indicate any other scheduled substance

3. No Analysis Performed

a. Drugs and Cannabis	Not Analyzed
	No Other Items Analyzed

4. Qualifiers/Inconclusive Findings

a. Drugs and Cannabis	Testing indicated the possible presence of XXX*, however, this was not confirmed.
	Testing indicates the presence of XXX*, however, this was not confirmed due to insufficient sample.
	Testing indicates the presence of XXX*, however, there is insufficient reference material available for identification at this time.
	Testing did not indicate a controlled substance
	Testing did not indicate a scheduled substance
	Testing did not indicate any other scheduled substance
	Testing performed; no conclusion drawn.

*XXX is the substance name.
†Y is the appropriate salt form or free base.

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APPENDIX II: DATA SUITABILITY

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APPENDIX II

Data, including instrumental data, observations, and calculations, obtained from analysis of an unknown sample must first be evaluated for suitability prior to drawing any conclusions utilizing the following suitability assessments. If a deficiency is indicated, mitigation steps will be necessary to allow for an appropriate and meaningful comparison to be made.

I. Definitions and Documentation

A. Data that is suitable for comparison meets the minimum standards and controls in addition to the requirements outlined in this Appendix.

1. Data will be included in the case file and documented in LIMS per the policies of each technique.

2. Data that provides information with limitations (e.g. indications on a GC-MS, lack of isomer differentiation, weak color tests, etc.) is still suitable. Results will be documented per the policies of each technique to reflect any limitations.

B. Data that does not meet the minimum standards and controls and/or the requirements outlined in this Appendix will be considered rejected data.

1. The decision to reject data will be made by the forensic scientist authoring the report.
    i. If the suggestion to reject data is made by a technical reviewer, this will be succinctly documented in the routing history through the rejection of the case.

2. For techniques which produce instrumental data, the data itself will be clearly identified as rejected and included in the casefile as delineated in the Quality Manual (QM-12).

3. For techniques which do not generate instrumental data, succinct documentation of the observations on the worksheet will suffice.

4. The reasoning for the rejection, along with the date the data was evaluated and deemed rejected, will be succinctly documented on the worksheet.

II. For Instrumental Techniques (GC-FID, GC-IR, GC-MS, AND FTIR)

A. Instrument Performance and Policy

1. Instrument acquisition failures, including missed injections and instrument stoppages, will be evaluated for their effect on data acquisition. In order to utilize acquired data for comparison, all associated data will meet the minimum standards

and controls applicable to the technique(s) employed. If the data does not meet established minimum standards and controls, or there is no data acquired, then the associated data will be rejected for comparison to a reference standard.

2. In order to utilize acquired data for comparison, all associated blanks and controls will meet the minimum standards and controls applicable to the technique(s) employed.
    i. If the blanks and controls do not meet the established minimum standards and controls, then the associated data will be rejected for comparison to a reference standard.

B. Detector Response

1. When using a technique that produces a detector response, the response will be at least three times the noise level to be considered a peak suitable for comparison to a reference standard. The short-term noise levels (those directly adjacent to the signal response) are used for determining response suitability. If a visual assessment does not clearly demonstrate a signal-to-noise ratio greater than 3:1, then a mathematical technique will be employed. (Use of chromatographic data processing software to determine signal-to-noise ratios is acceptable.)
    i. If the response is not at least three times the noise level to be considered a peak, the data will be rejected for comparison to a reference standard.

C. Chromatographic Peak Shape

1. Acceptable peaks are typically Gaussian, but slight asymmetry and tailing is normal with temperature-gradient methods. In the event that a chromatographic peak is not Gaussian, the analyst will evaluate the level of fronting or tailing to determine the significance of the irregularity.

2. The peak of a single analyte usually produces a single apex to be used for comparison. In the event of a split peak, a single distinct apex which is at least two times the height of any secondary apex not baseline resolved, may be used for comparison to a reference standard.

    i. GC-FID: If the peak shape is considered unacceptable, the data will be rejected for comparison to a reference standard and mitigation steps will be taken upon reanalysis of the sample.
    ii. GC-MS: If the peak shape is considered unacceptable for retention time comparison, spectral data may still be utilized for comparison to a reference standard.

D. Resolution

1. If multiple peaks are present, chromatographic resolution shall be sufficient to evaluate a substance with minimal interference from other substances or impurities. Resolution will also be sufficient to differentiate between IR bands and mass assignments. Analysts will assess the extent to which any potential overlap affects identification of a specific analyte to the exclusion of interferences as follows.
    i. Baseline resolution between multiple peaks is recommended. At minimum, adjacent chromatographic peaks must have discernable apexes.
    ii. Data processing procedures can be used (e.g., spectral subtractions, extracted ion chromatogram, etc.).
    iii. If the resolution is not sufficient to show separation, then the associated data will be rejected for comparison to a reference standard.

III. For Non-Instrumental Techniques (TLC, Color Tests)

A. Blanks and Controls

1. Required blanks and controls will meet the minimum standards and controls applicable to the technique(s) employed to utilize the associated data for comparison.
    i. If the blanks and controls do not meet the established minimum standards and controls, then the associated data will be rejected for comparison to a reference standard.

B. Color Tests

1. Visually assess the results for the presence or absence of positive test indications (color changes) as defined by minimum standards and controls or in comparison to a primary reference standard.
    i. The absence of response will not be used as a preliminary test for the positive identification of a controlled substance.
    ii. Reasons for color test data to be rejected may include:
        a. a reagent batch is expired
        b. the reagent fails quality checks
        c. a substance known to inhibit color is present in the sample (e.g. tableting dyes, DMSO, etc.)

C. Thin Layer Chromatography

1. Visually assess the plate for the presence or absence of positive test indications (spot appearances) as defined by minimum standards and controls.
    i. If the reference materials fail to produce data, the plate will be rejected.

2. The spots of the target analyte and the reference standard(s) are suitable for comparison when they are approximately circular or a rectangular band (not a

streak), adequately resolved from other sample components as well as other standards included on the plate, and visible when exposed to UV light or a chemical visualizing reagent.

i. If the spots fail to meet the above criteria, the data will be rejected.

Note: Concentration disparities and their impact on data suitability

Characteristics that may be affected by concentration disparities may include chromatographic peak shape, retention time, mass spectral ion ratios, TLC spot size and shape, and intensity of any color reactions. While concentration disparities alone are not a reason to reject data, their impact may be cause for a rejection. Mitigation steps for many issues often begin with an assessment of the concentration of a sample. When experimental conditions, including concentrations, between the sample and the reference standard are similar, meaningful comparisons are more likely.

MINIMUM STANDARDS AND CONTROLS FOR TESTING TECHNIQUES

Minimum standards and controls for each procedure or testing technique can be found in the relevant section of the Procedures Manual.

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APPENDIX III: EXTRACTION, SEPARATION, AND DERIVATIZATION METHODS

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APPENDIX III
EXTRACTION AND SEPARATIONS

INTRODUCTION

The purpose of an extraction is to separate drugs from a matrix or other drugs. The matrix can be a variety of materials including, but not limited to, a liquid, solid, pharmaceutical unit, or food. Since the process of manufacturing both legitimate pharmaceuticals as well as clandestine street drugs can vary significantly, it would be impossible to create an exhaustive list of all possible extractions. Therefore, this policy presents general guidelines and examples of the types of potential extractions available.

SAFETY CONSIDERATIONS

Standard laboratory practices involving the use of solvents, acids, and bases.

PREPARATIONS

Use the following table to assist in the preparation of the various reagents:

Definitions

Formality (F)	$\frac{\text{gram formula weight}}{\text{liter}}$	The number of formula weights of solute in one liter of solution.
Molarity (M)	$\frac{\text{moles}}{\text{liter}}$	The number of moles of solute in one liter of solution.
Molality (m)	$\frac{\text{moles}}{\text{kilogram}}$	The number of moles of solute of per kilogram solvent.
Normality (N)	$\frac{\text{equivalents}}{\text{liter}}$	The number of equivalents of solute in one liter of solution.
Weight %	$\frac{\text{weight of solute x 100}}{\text{weight of solution}}$	Concentrations of commercial aqueous reagents are weight %.
Volume %	$\frac{\text{volume of solute x 100}}{\text{volume of solution}}$	
Weight Volume %	$\frac{\text{weight of solute mg x100}}{\text{volume of solution, mL}}$	Weight volume percent is used to indicate dilute aqueous solutions of solid reagents.

Stock Chemicals:

The composition of some inorganic acids and bases as supplied by vendors (either as concentrated aqueous solutions or as solids) are given below:

Acid/Base	Formula Weight		Molarity	% by Weight
Acetic Acid (CH_3COOH)	60.05		17.5	99-100
Ammonium Hydroxide (NH_4OH)	35.05		7.4	28-30
Hydrochloric Acid (HCl)	36.46		12.0	36.5-38
Nitric Acid (HNO_3)	63.02		16.4	69
Phosphoric Acid (H_3PO_4)	98.00		14.7	85
Potassium Carbonate (K_2CO_3)	138.21		---	---
Sulfuric Acid (H_2SO_4)	98.08		18.0	95-98
Sodium Bicarbonate ($NaHCO_3$)	84.02		---	---
Sodium Carbonate (Na_2CO_3)	105.99	(anhy)	---	---
Sodium Hydroxide (NaOH)	40.01		---	---

Preparation of Reagents

The following charts can be used as a reference for the preparation of various concentrations (dilutions) of acids and bases:

Liquid Acid/Base Reagents	Desired Concentration	Quantity of Concentrated Acid/ Base Diluted to 1 Liter
Acetic Acid	3N	172 mL
Ammonium Hydroxide	3M, 3N	405 mL
Hydrochloric Acid	3N	250 mL
Hydrochloric Acid	0.1N	8 mL
Hydrochloric Acid	15% (4.4M)	367 mL
Nitric Acid	3N	183 mL
Phosphoric Acid	3M, 9N	205 mL
Sulfuric Acid	3M, 6N	168 mL
Sulfuric Acid	6M, 12N	336 mL
Sulfuric Acid	0.1N	2.8 mL

Solid Basic Reagents	Desired Concentration	Quantity for Dilution to 1 Liter
Potassium Carbonate	5%	50 g
Sodium Bicarbonate	1M	84 g
Sodium Carbonate	1.5M, 3N	156 g
Sodium Carbonate	2%	20 g
Sodium Hydroxide	0.5N	20 g
Sodium Hydroxide	2N	80 g

INSTRUMENTATION

None Required.

MINIMUM STANDARDS AND CONTROLS

1. All extractions will be recorded on the worksheet.

2. Stock bottles of dilutions/reagents are to be tested when prepared and as indicated in QM-14, using pH paper or another appropriate test. A logbook (or online equivalent) will be kept of the testing to include at least the name (or initials) of the preparer of the reagent, the date the reagent was authenticated (or re-authenticated), and the expiration date of the reagent. Any manufactured reagents/dilutions will be tested when opened and as indicated in QM-14; if no manufacturer's expiration date is given, it will be assigned a one year date and treated in the same manner as in-house reagents/dilutions.

3. All solutions utilized for extractions and separations will be labeled according to QM-14, including a hazard label.

PROCEDURE OR ANALYSIS

Solvent Extractions

Solvent extractions (dry extractions) are based on the differences in solubilities between substances.

Dry extractions involve washing a sample with a solvent in order to isolate a compound of interest from a mixture. If the desired component is soluble in the solvent and the other components are not, the solvent can be separated from the insoluble, either by filtering or using a centrifuge. This solvent is then dried down to yield the desired component. Conversely, a solvent may be selected to wash away unwanted soluble components from a mixture, leaving behind the insoluble compound of interest.

	Chloroform	Methanol	Ether	Acetone
Acetaminophen	v sl sol	sol	insol	sol
Aspirin	sol	sol	sol	sol
Amphetamine HCl	sol	sol	insol	sl sol
Amphetamine SO4	insol	sl sol	insol	
Benzocaine	sol	sol	sol	
Caffeine	sol	sl sol	sl sol	sol
Cocaine HCl	sol	sol	insol	insol
Cocaine free base	sol	sol	sol	sol
Codeine	sol	sol	sl sol	insol
Codeine Phosphate	insol	sl sol	insol	
Diazepam	sol	sol	sl sol	
Diphenhydramine HCl	sol	sol	insol	
Ephedrine	sol	sol	sol	sol
Ephedrine HCl	v sl sol	sol	insol	
Heroin HCl	sol	sol	insol	
Heroin	sol	sol	insol	
Lidocaine	sol	sol	sol	
Lidocaine HCl	sl sol	sol	insol	

Methamphetamine HCl	sol	sol	insol	insol
Nicotinamide	sl sol	sol	sl sol	
Procaine	sol	sol	sol	
Procaine HCl	sl sol	sol	insol	
Tetracaine	sol	sol	sol	
Tetracaine HCl	sl sol	sl sol	insol	insol

A variation of the solvent wash procedure is to use acid or base washed solvents. One part acid or base is added to ten parts solvent.

Acid/Base Extractions (Liquid/Liquid)

The liquid/liquid extraction uses two immiscible solvents. Water or aqueous acid/base and immiscible organic solvents are normally used. The desired component is partitioned into one solvent while the other components are partitioned into another solvent. Separating the two phases will yield the desired substance.

Note: Chloroform is best for general screens. Fewer drugs extract into hexane, however it usually yields a cleaner sample.

Note: If the substance being extracted is volatile (such as methamphetamine) concentrated HCl gas should be bubbled through the solvent layer before evaporation to yield the insoluble hydrochloride salt.

EXAMPLES OF ACID/BASE EXTRACTIONS

Amphetamine	1. NaOH, hexane. Bubble HCl through hexane.
Chlordizaepoxide	1. NaOH, chloroform. Petroleum ether recrystallization.
Cocaine	1. NaOH, hexane. 2. Wash with chloroform and pet ether mixture.
Codeine	1. NH_4OH, chloroform. 2. Acetone wash, dry insolubles, add water, then K_2CO_3 and hexane (separates out acetaminophen or aspirin).
Diazepam	1. HCl, chloroform. 2. Chloroform, pet ether, or acetone wash. 3. NaOH, chloroform.
Dihydrocodeine	1. K_2CO_3, hexane. Petroleum ether recrystallization.
Ephedrine	1. NaOH, hexane. Bubble HCl through hexane.
Heroin	1. 3N HCl, chloroform.

Hydromorphone	1. NH_4OH, chloroform.
LSD	1. Water, NH_4OH, chloroform.
Methamphetamine	1. NaOH, hexane. Bubble HCl through hexane. 2. Wash with acetone. To insolubles, add chloroform: pet ether or NaOH, hexane (with HCl bubbled through). This separates ephedrine which is soluble in acetone.
Morphine	1. Concentrated NH_4OH, warm chloroform: isopropanol (3:1).
PCP	1. HCl, chloroform. 2. NaOH, hexane. 3. Chloroform wash.
Propoxyphene	1. H_2CO_3, hexane. Petroleum ether recrystallization. 2. K_2CO_3, hexane. Bubble HCl through hexane. Discard hexane. Wash insoluble white powder with hexane, discarding solvent. To the white powder add K_2CO_3, hexane. Dry hexane, petroleum either recrystallization.

REFERENCES

1. Drug Chemistry Training Manual - Section DC-IA-6.

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DERIVATIZATION

INTRODUCTION

Derivatization is used to reduce or eliminate the problems of activity related to any hydroxyl (free, or existing as part of a carboxylic acid, e.g. GHB), or amine group (e.g. d,l-amphetamine, d,l-methamphetamine). These functional groups usually cause peak tailing or loss of response due to the interactions of the functional groups with liners, or active sites on the columns. This activity can be reduced or eliminated by chemically converting active groups to inactive species. Derivatizing the sample prior to using a gas chromatograph or mass spectrometer would overcome problems in identification and characterization.

SAFETY CONSIDERATIONS

Standard laboratory safety procedures for handling corrosive materials and solvents. Special handling requirements are included with the material safety data sheets and should be read prior to the use of these derivatizing agents.

INSTRUMENTATION

Gas Chromatography with Flame Ionization Detection or Gas Chromatography-Mass Spectrometry

MINIMUM STANDARDS AND CONTROLS

1. The derivatizing methods will be noted on the worksheet.
2. A standard will be run using the same derivatizing method used on the sample to establish reference data and to authenticate the reagents. Any manufactured reagents will be tested when opened and as indicated in QM-14; if no manufacturer's expiration date is given, it will be assigned a one year date and treated in the same manner as in-house reagents.
3. Refer to the standards and controls associated with the instrumental procedures of choice (GC-FID or GC-MS).

PROCEDURE OR ANALYSIS

The following procedures or analyses are examples of derivatization methods available. For all methods the following procedures should be noted:

A. Store unused derivatizing agent in the refrigerator capped and sealed to protect from moisture.

B. The derivatizing agent must be at room temperature before use.

1. Intended Use: d,l-amphetamine, d,l-methamphetamine

Reagents: (S)-(-)-N-(Trifluoroacetyl) prolyl Chloride (purchased as 0.1 M solution in dichloromethane)
Conc. Hydrochloric Acid
Methanol

1% HCl in Methanol: Add one milliliter of concentrated HCl to 99 mL of Methanol.

Procedure:

1. For a powdered sample, add several milligrams to screw top test tube.
2. Add ½ mL of derivatizing agent to the tube.
3. Place the tube in a heating block (setting of 5 or 6 about 65°C) for 10 to 15 minutes.
4. Remove from heat and, if necessary, dilute with dichloromethane.

Notes:

1. If the original sample is a liquid, e.g. after extraction ½ mL of 1% Methanolic HCl can be added to reduce evaporation of amines. Take this to dryness before derivatizing.
2. If the sample is a free base liquid the derivatizing agent can be added directly.
3. Do not use methanol or ethanol as solvents since they can be derivatized (Step 4).
4. Store unused derivatizing agent in the refrigerator capped and sealed to protect from moisture.
5. The derivatizing agent must be at room temperature before use.

For comparison purposes, a standard should also be derivatized. Once the sample is derivatized, it can be used with the GC-FID or GC-MS. The d and l derivatives of Amphetamine and Methamphetamine can be distinguished by retention time.

2. Intended Use: GHB

Note: Due to the absence of the hydroxyl substituent on GBL, it is not derivatized during the reaction.

Reagents:
Dichloromethane (CH_2Cl_2)
Bis(trimethysilyl) trifluoroacetamide (BSTFA)
Bis(trimethysilyl) trifluoroacetamide (BSTFA) with 1% trimethylchlorosilane (TCMS)

Procedure:

1. Use 5 mL of sample.
2. Add 5 mL CH_2Cl_2 vortex, remove organic layers and place into a clean test tube.
3. Dry down organic layer (no heat).
4. Add 100 microliters of BSTFA and cap tightly (sample is volatile).
5. Place in a 70°C oven for 30 minutes.
6. Place into a microvial and inject on GC-FID or GC-MS.

REPORT WORDING

See Appendix I - Report Wording.

REFERENCES

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DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX IV: CHEMISTRY MINIMUM STANDARDS AND CONTROLS

SECTION A: ADMINISTRATIVE POLICIES APPLICABLE TO ANY ANALYSES

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APPENDIX IV

Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete an identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

ADMINISTRATIVE POLICIES APPLICABLE TO ANY ANALYSES

A. Tests and Controls

1. Routine Formulations, Standard Tests - There is no need to include how tests are performed in the case file, as long as they are standard tests performed routinely. The same reasoning applies to formulations of reagents, solutions, and standard in-house methods. Many of these will be found in the chemistry section procedures manual or a laboratory's chemistry section manual.

2. All reference spectra /chromatograms used for identification shall be included in the case file.

3. GC-FID and GC-MS Blank Policy

   The blank serves two primary functions: first, as a quality check on the chemicals used during the sample preparation process and second, as a quality assurance measure for monitoring laboratory technique and instrumentation. For the purposes of this policy, lab case item or item refers to the parent item received from an agency that may include one or more unknown units for analysis. Unit(s) refers to the unknown object(s) requiring analysis received within the item. Sample(s) refers to the analyzed portion(s) of the unit.

   a. When blanks need to be run:

      Prior to the sample run, at least one blank will be injected per lab case item under the same conditions and method as the sample run(s). All consecutive blanks between samples in multiple unit cases will be run using the same conditions and method as the sample runs. This includes all instrument parameters from the injection to detection. In cases where a trace or residue amount of a substance is suspected, a blank will be run immediately before the suspected trace or residue sample. If multiple units are analyzed, no more than ten samples will be run after a single blank.

   b. Preparation of blanks:

      The blank will be prepared in the same manner as the sample. This includes all solvents and reagents, including those for extraction and derivatization. Any solvent other than methanol must be noted on the worksheet. If the sample is

diluted in an internal standard solution, the blank will be prepared from the internal standard solution. If the sample is extracted, the blank will be extracted. If the sample is concentrated prior to injection, the blank is to be concentrated as well. In such cases that a neat sample must be injected directly, the solvent chosen for the blank should be documented on the worksheet.

c. Blank acceptance:

If the blank has a peak at the same retention time as the target compound, the blank will be further evaluated for acceptability. For GC-MS, noise that is attributed to column/septa bleed and/or peaks that are attributed to solvent components do not invalidate the blank. It is also acceptable if the blank has peaks at retention times other than the target compound. For GC-MS chromatograms, any peak(s) present will be checked and documented in the case file. If the blank is determined to be unacceptable, the blank and unit(s) will be run again. Raised gas chromatograph baselines also do not invalidate the blank.

d. Documentation:

Blanks will be included in the working file and will be noted on the worksheet. In multiple unit cases, all consecutive blanks run between samples will be included. Each blank for a case must be labeled in a way that specifies when (e.g., timestamp) it was run in relation to the sample(s). Labeling blanks with the item number is optional.

B. Evidence Handling Procedures

These are general procedures for the handling of Chemistry evidence.

1. Seals

a. Whenever possible, all evidence is to be sealed when received, when placed in the vault and when not actually being analyzed. If possible, handle evidence in a manner which preserves the integrity of the material inside and does not destroy or alter existing seals.

b. After analysis, seals on internal packaging are only necessary to preserve the condition of the evidence (to prevent leakage or cross-contamination). All external packages shall be sealed. The minimum marks needed on these exterior seals are initials and date.

2. Markings

a. All external packages of evidence will contain the case number, item number, initials and date. After analysis, internal packaging will be marked with a

minimum of the item/subitem number in a manner that allows for individual identification as reflected on the worksheet. Any additional markings can be at the discretion of the analyst. The internal packaging will then be sealed into an outer package that is marked with the case number, item number, date and initials.

C. Analysis of Evidence

1. When conducting casework, all observations, examinations and conclusions must be recorded and conducted in accordance with Command Directives guidelines.

2. In general, it is not necessary to conduct an analysis on each item submitted. Evidentiary value and agency needs should determine the extent of analysis.

3. If possible, no more than half the evidence shall be consumed in analysis.

4. Every attempt shall be made to preserve evidence. When the entire sample is used, extracts must be returned to the case.

5. Per Command Directives ESH Appendix 16, field test kits are not to be submitted as evidence. If a field test kit is found within submitted evidence, it will be packaged to prevent contamination of other evidence and returned to the agency. DO NOT DISCARD!

D. Mass Determination

1. Balances will be checked weekly using masses across the range of the balance, including those listed in QM-11, according the table below.

Balance Capacity	Weights
200 g, 400 g	0.1 g, 1 g, 10 g, 200 g
500 g, 610 g	0.1 g, 1 g, 10 g, 500 g
4,000 g	0.1 g, 1 g, 10 g, 2,000 g
6,000 g, 8,000 g	500 g, 1,000 g 5,000 g
10,000 g, 32,000 g, 150,000 g	500 g, 5,000 g, 10,000 g

The examiner's identity, date, and exact masses obtained will be recorded in a logbook (or online equivalent). Balances not used routinely will be checked monthly. A balance check will be effective for seven days. Each laboratory will have a policy to follow should a balance fail to pass a standardized weight check.

a. Balances and weight sets will be calibrated by an ISO certified vendor on a routine basis as required by QM-11.

2. The net mass of all substances will be determined prior to analysis with the following exceptions:

    a. Samples involving trace or residual amounts of material.

    b. Instances in which gross/estimated mass determination of numerous samples indicates a mass less than legal limits as set by statute(s).

    c. Instances in which the net or gross/estimated mass far exceeds the maximum legal limits mass as set by statute(s). In this instance, a sample with net mass exceeding the legal limit would suffice.

    d. Samples involving pharmaceutical preparations where preliminary identification indicates a non-scheduled substance.

    e. Samples involving pharmaceutical preparations where preliminary identification indicates a lower scheduled substance than a previously identified higher schedule substance.

    f. Cases involving the submission of individual suspected Cannabis plants or the submission of representative samples of individual suspected Cannabis plants where the agency plans to pursue charges based on the number of plants present, not the weight.

    g. For cases involving large amounts of evidence (e.g. powders, chunky substances, etc.), a gross weight will be obtained and the evidence will be screened. Instances where indications of a highly potent substance that may pose a risk to the analyst's safety are present (e.g. carfentanil), no further weighing will take place.

    h. The vessel used to weigh the evidence will not exceed the size/boundaries of the balance pan.

        a. If a single unit (i.e. one large bundle of plant material) exceeds the size/boundaries of the balance with the largest pan, the unit will be weighed in portions. Each weight will be recorded and added together for a total net weight of one unit. Justification for the division will be documented on the worksheet.

3. The balance reading should be recorded using the following conventions:

    a. The mass will be reported to the 0.1 gram decimal place when the balance used permits. Never round off the balance readings. Record all numbers read from the balance on the worksheet. For the report, the additional digits are dropped e.g, a balance reading of 553.1 grams will be reported as 553.1 grams, a balance reading of 10,352 grams will be reported as 10,352 grams and a balance reading of 1.341 grams will be reported as 1.3 grams. The mass will be reported out to the crucial

figure when needed for charge determination. At a weight class, truncation occurs after the final digit significant to the charge determination e.g., a balance reading of 1.008 grams will be reported as 1.008 grams for a 1 gram weight class and a balance reading of 1.032 grams is reported as 1.03 grams for a 1 gram weight class.

b. Less than one gram is singular and shall be preceded by a zero to indicate the placement of the decimal. Example: 0.1 gram. Note: One gram is included as singular.

c. If the mass is less than 0.1 gram, it shall be reported as "less than 0.1 gram". For smaller amounts, an option is to report it as a residue.

d. If the charge is affected in a multiple item, truncating must occur after the addition of the masses of the items.

e. Rolled cigarettes/cigars are weighed in their entirety. If a filter is present, remove it prior to mass determination. The plant material is not separated from the paper.

f. Legitimate pharmaceutical capsules are weighed in their entirety. For clandestine capsules, the capsule material is to be treated as packaging. An exception can be made for substances that are subject to a dosage unit count in Illinois statutes.

4. Measurement uncertainty will be calculated per Appendix V.

E. Sampling

1. When possible, the analyst shall take a representative sample on which to perform tests.

2. It is not necessary to test every item in a case in order to make a determination about the presence of Cannabis or a Controlled Substance. Sample requirements for multiple item cases are listed in the Sampling Plan under Section H (DC-H-1).

F. Worksheet

1. The worksheet shall be a summary of all tests done and their results.

a. Identification of the instrument and balance used in the analysis will be noted in the case records

2. On each worksheet, the following information must be found:

a. The case number assigned to a particular item of evidence.

b. The item number.

c. The initials or signature of the analyst.

d. The date the worksheet was completed.

e. A header which includes "Illinois State Police, Division of Forensic Services, Forensic Sciences Command."

3. Included on the worksheet should be the following:

a. A description of that item to include packaging, repackaging (if different than original), and a physical description of the contents.

b. All tests and measurements performed on the item, including their results, and any of the information required elsewhere under minimum standards and controls.

c. The conclusions reached by the analyst.

G. Reference Materials and Collections

1. Reference Materials

A. In-house Drug Standards (Primary and Secondary)

1. The name of the drug, date received, the source, and lot number, will be recorded for all controlled drug standards and the record will be revised as drugs are received or consumed. If a quantitative drug standard is to be stored, the expiration date and/or re-authentication date will also be recorded.

2. When first used, IR, MS, or other approved techniques will be run, and the spectrum will be used to authenticate and verify the identity of the drug with literature and/or in-house standards.

a. For qualitative standards, no expiration dates will be assigned. Some standards might have dates assigned by the manufacturer. The continued use of the standards in obtaining IR and MS data serves to verify that the standards are fit for use. As long as the data obtained from drug standard can serve to verify the identity of the standard, it can be used past any manufacturer supplied expiration date.

b. Drug standards that are used in quantitative work will be assigned an expiration date if not supplied with one by the manufacturer. The date will not be more than five years from the date of receipt. All quantitative drug standards can be re-authenticated; the newly assigned expiration date will not be more than 5 years from the date of re-authentication.

c. Any standard that is deemed to be unfit for use will be discarded.

3. Primary standards are defined as those obtained from legitimate sources. Secondary standards are those obtained from other sources and verified in-house; these standards should be kept to a minimum.

4. All controlled standards (primary and secondary) will be inventoried yearly. Those over 1 gram will be weighed and recorded. Documentation of the annual inventory will be recorded in LAM.

B. Literature Sources and External Standard References

1. If no in-house drug standard is available for comparison, literature and external sources may be referenced. In determining the legitimacy of external sources, analysts should consider the credibility and qualifications of the author, publication and format of the reference (e.g. scholarly journals or internet sites), date of publication or acquisition, and accompanying documentation (method parameters, lot numbers, etc.).

2. In general, library searches should not be considered legitimate references for data comparisons. Exceptions include peer-reviewed and published libraries or libraries created within the Command using traceable reference materials. Any library reference meeting these exceptions must be evaluated for legitimacy and relevance similar to any literature or other external reference.

C. Other Reference Materials

1. PFTBA – This reference material is used to tune a mass spectrometer. PFTBA will be obtained from ISO accredited vendors. Appropriate documentation, such as a certificate of analysis, should be retained when available. The process of tuning the mass spectrometer will serve to authenticate the PFTBA reference material. Since the tune is verified every time it is performed, the PFTBA can be used past a manufacturer provided expiration date.

2. Polystyrene – This reference material is used during the function check for infrared spectrophotometers. It must be a traceable standard, from an ISO accredited vendor, with appropriate documentation. If the standard is provided with a manufacturer's expiration date, the standard may be re-authenticated to extend the date in five year intervals. The new date and analyst's identity should be included on the standard or in the logbook (or online equivalent).

2. Reference Collections

A. In-house spectral libraries

1. All spectra included in such collection will be uniquely identified as to the substance, its source (lot number or other identifier), and to the instrument on which it was acquired.

2. The collection will be kept within the Drug Chemistry section.

3. The collection can be archived as needed according to local procedures.

B. Sample collection for reagent testing

1. A collection of known materials may be kept for the purposes of testing reagents (color tests, TLC visualization sprays, etc.)

2. These materials may be purchased or samples taken from casework.

3. Each item in the collection will be uniquely identified.

4. Access to the collection will be limited, and use will be limited to the testing of reagents.

H. Temperature Controlled Environments

1. Temperature Checks
   a. A temperature check of the refrigerator and/or freezer will be completed monthly and recorded in LAM.
      1. Refrigerators must be kept between 2°C and 10°C (between 35°F and 50°F).
      2. Freezers must be kept between -25°C and -10°C (between -13°F and 14°F).
   b. If the temperature is out of the acceptable range, adjust the temperature dial if necessary and recheck.
      1. If the temperature cannot be maintained within the specified range listed above for a period of 24 hours, all contents will be relocated to an appropriate working refrigeration unit. The refrigerator or freezer that was out of the acceptable temperature range will be placed out-of-service until it can be repaired or replaced.
2. Thermometers
   a. Only thermometers with ISO/IEC 17025 Certificate of Calibrations may be used. These thermometers must be traceable to national or international standards (e.g. NIST). The calibration certificate for each thermometer will be entered in the LAM. The vendor's calibration serves as the performance check. The thermometer may be placed into use immediately and used until the expiration date on its certificate.

I. Abbreviations

The following are the abbreviations allowed for use in LIMS. They can be used as written or with any variation in case type (upper or lower).

1. General

a.	ASA	Assistant State's Attorney
b.	bl	blank
c.	BSB	background subtraction
d.	c/ or cont	contains/containing
e.	CIA	consistent in appearance
f.	Cland	clandestine
g.	Conc	concentrated
h.	Dist	distance
i.	EIC	extracted ion chromatogram
j.	est	estimated
k.	ET	evidence tape(d)
l.	Evap	evaporated
m.	Exp	expected
n.	Expr	Expiration/expire(d)
o.	Ext	extraction
p.	FID	Flame ionization detector
q.	Func	function
r.	GC	Gas chromatograph
s.	GW	gross weight
t.	HS	heat sealed
u.	ISP	Illinois State Police
v.	Inc	included
w.	Ind	indicates/indicated
x.	Inv	inventory
y.	LAM	Laboratory Asset Manager (module in the LIMS)
z.	LIMS	Laboratory Information Management System
aa.	mL	milliliter
bb.	MS	Mass spectrometer
cc.	MU	Measurement Uncertainty
dd.	NA	Not Analyzed
ee.	NR	No Reaction
ff.	NS	not scheduled/non scheduled
gg.	NSSF	No scheduled substance found
hh.	NSSI	No scheduled substance indicated
ii.	nw	net weight
jj.	PD	Police Department
kk.	pg	page
ll.	pharm	pharmaceutical
mm.	prep	prepared
nn.	quant	quantitation
oo.	reaut	Reauthentication

pp.	RT	retention time
qq.	SA/SAO	State's Attorney/State's Attorney's Office
rr.	SDT	Subpoena Duces Tecum
ss.	SN	Serial number
tt.	STD	standard
uu.	TIC	total ion chromatogram
vv.	Unk	unknown
ww.	USC	United States Currency
xx.	Wt	weight

2. Scientific

a.	CHCl3	chloroform
b.	D/L	Duquenois-Levine
c.	g	gram
d.	HCl	hydrochloric acid
e.	M	molar
f.	MeOH	methanol
g.	N	normal

Notes may also contain instrument names, drug names, chemical formulas, generally accepted nomenclature, and routinely used scientific abbreviations. The list above includes examples but is not a fully comprehensive list.

3. Symbols

a.	#	number
b.	$	dollar
c.	& or +	and
d.	<	less than
e.	>	greater than
f.	~	indicates
g.	µ	micro
h.	x	times
i.	?	indicates/unknown
j.	✓	checked/ok; looked at
k.	-	subtracted

MINIMUM STANDARDS AND CONTROLS FOR TESTING TECHNIQUES

Minimum standards and controls for each procedure or testing technique can be found in the relevant section of the Procedures Manual.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX IV: CHEMISTRY MINIMUM STANDARDS AND CONTROLS

SECTION B: ADMINISTRATIVE POLICIES FOR CANNABIS ANALYSIS

Reviewed by:

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Forensic Scientist Adrienne A. Hirsch, Chairperson
Drug Chemistry Command Advisory Board

Approved by:

_____

Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

APPENDIX IV-B

Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete an identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

ADMINISTRATIVE POLICIES FOR CANNABIS ANALYSIS

The Illinois Cannabis Control Act (CCA), Chapter 720, Act 550 and the Cannabis Regulation, Tax Act (CRTA), Chapter 410, Act 705, and the Industrial Hemp Act, Chapter 505, Act 89 of the Illinois Compiled Statutes contain the regulations associated with the control of Cannabis.

While the protocols, methods and procedure give directions for the analysis of Cannabis, there are some specific administrative procedures directly applicable to Cannabis analysis. These various procedures are listed in this appendix.

The following are procedures associated with the identification of Cannabis:

A. Positive Identification

1. To identify plant material as Cannabis, two positive tests are acceptable if one of the tests provides structural confirmation (e.g. GC-MS). Additional tests may be done at the discretion of the analyst. The tests used for Cannabis analysis are listed below:

    a. Duquenois-Levine (D-L) test – See DC-IIIA-2.

    b. FTIR – See DC-F-1.

    c. GC-FID – See DC-D-1.

    d. GC-IR – See DC-I-1.

    e. GC-MS – See DC-G-1.

    f. Macroscopic Examination – See DC-IIIA-1.

    g. Microscopic Examination – See DC-IIIA-1.

    h. TLC - See DC-C-1.

The macroscopic and microscopic examinations, when performed together, count as one test. It is permissible to perform a microscopic examination in conjunction with another test that provides structural confirmation, however, it is not sufficient to perform the macroscopic examination with any other technique, even if the second technique provides structural confirmation.

2. It is not necessary to test every item in a case in order to make a determination about the presence of Cannabis. Sample requirements for multiple item cases are listed in the Sampling Plan under Section H.

3. Cases found to contain Cannabis will specify the cannabinoid identified on the report.

4. Other tests may be submitted at the discretion of the chemist as long as they meet the minimum standards and controls guidelines.

B. E-cigarette cartridges

Because of the level of difficulty associated with opening e-cigarette cartridges, as well as the sheer volume necessary to meet weight class limits, when one cartridge has been analyzed and found to contain a cannabinoid, no others will be tested. If the one analyzed cartridge does not contain a cannabinoid, the analyst will use his or her discretion to determine if more items need to be analyzed. Further analysis on any remaining cartridges submitted will be at the discretion of laboratory management and only completed if requested by the State's Attorney's Office. The unanalyzed cartridges will NOT need a gross or estimated weight. They will be reported as cartridges (additional description optional) and a conclusion of "Not Analyzed".

C. Not Cannabis or No Cannabis Found Determination

At least two negative tests are required to eliminate the possibility of the presence of Cannabis. When "Not Cannabis" or "No Cannabis Found" is the only determination made for a piece of evidence, one of these must be an instrumental test that also eliminates the possibility of other controlled substances, including high molecular weight synthetic cannabinoids. It is not necessary to report "Not Cannabis" or "No Cannabis Found" when another substance is identified.

D. No Analysis Determination

When identifying Cannabis, the entire amount submitted does not need to be analyzed. Analysis need only be conducted to the weight limits for Illinois penalties.

MINIMUM STANDARDS AND CONTROLS FOR TESTING TECHNIQUES

Minimum standards and controls for each individual procedure or testing technique can be found in the relevant section of the procedures manual.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX IV: CHEMISTRY MINIMUM STANDARDS AND CONTROLS

SECTION C: ADMINISTRATIVE POLICIES FOR CONTROLLED SUBSTANCE ANALYSIS

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Approved by:

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Forensic Sciences Command

APPENDIX IV

Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete an identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

ADMINISTRATIVE POLICIES FOR CONTROLLED SUBSTANCE ANALYSIS

The Illinois Controlled Substances Act governs most substances controlled in the state of Illinois. The act is found in Chapter 720, Section 570 of the Illinois Compiled Statutes. Substances controlled via administrative rule are listed separately in the Illinois Administrative Code, Title 77, Chapter X, Subchapter e, Part 2070. Additionally, the Kratom Control Act, The Methamphetamine Control and Community Protection Act, and the Methamphetamine Precursor Control Act are of interest to the Drug Chemistry Section. These can be found in Chapter 720, Sections 642, 646, and 648 respectively of the Illinois Compiled Statutes.

While the protocols, methods and procedures give directions for the analysis of suspected controlled substances, there are many administrative procedures which must be addressed and which do not lend themselves to a specific analysis. These various procedures are collected into this appendix.

The following are procedures associated with the identification of any substances, regardless of their control status:

A. Positive Identification

1. There are minimum tests necessary for the identification of a drug. Two tests must be used to confirm the presence of a drug. One test will include IR, MS or another approved technique providing structural confirmation. The other test will be an approved preliminary testing technique or acceptable instrumental screening test in which the drug is indicated.

   It is acceptable to use two mass spectra to meet the minimum test requirements only if the following conditions are met:

   a. The second mass spectrometer is different from the first AND there is a column phase change.

   b. When a purchased reference standard is available and the sample warrants use of two mass spectra (e.g. residues, complex mixtures, synthetic drugs), the mass spectral data will be used **and, if they are the only tests used to make a determination,** a documented comparison of retention time data for at least one of the runs will also be utilized.

c. When purchased reference standards are unobtainable, the results of two GC-MS runs will be compared to two literature sources and documented in the case file. Therefore, these cases are exempt from the retention time comparison requirement.

Additional tests will be done at the discretion of the analyst.

2. It is not necessary to test every item in a case in order to make a determination about the presence of a controlled substance. Sample requirements for multiple item cases are listed in the Sampling Plan under Section H.

3. Any specialized analytical procedures used will be documented in the laboratory's chemistry section manual or on the worksheet.

B. No Scheduled Substance Found Determinations

1. At least two different tests, excluding color tests, will be used that together eliminate the possibility of the sample containing a controlled substance.

    a. Two GC-MSs must only be used when the sample warrants it (e.g. residues, complex mixtures).
        i. The second mass spectrometer is different from the first AND there is a column phase change.
        ii. Retention time data must be taken into consideration and documented in the case file. When it is not appropriate to make a retention time comparison to a standard, e. g. no peaks are present, peaks do not meet acceptance criteria or peaks present do not correspond to a controlled substance, the reason the comparison was not performed will be documented. This can be documented as "No relevant peaks for retention time comparison" or "No peaks of interest for retention time comparison" or other wording that acknowledges why the retention time data could not be taken into consideration.

2. A determination of no scheduled substance found requires one test to be a general screening run on a GC-MS instrument.

3. When testing shows possible indications of a substance of interest, for example through weak color tests or a mass spectrum with the presence of major ions associated with a possible controlled substance, it is necessary for the analyst to document an attempt to increase the sensitivity of the analytical process. This can be achieved in the following ways, often resulting in additional testing beyond the minimum two tests required:

    a. An increase in sensitivity through sample preparation (e.g. use of more sample, extraction and concentration to a microvial) or

b. An increase in sensitivity through instrumental techniques (e.g. lower split ratio, increased injection volume, manual injections)

4. A determination of no scheduled substance found can be made without excluding all possible controlled substances. Unless additional information is provided, only those drugs normally encountered in a laboratory need to be eliminated.

5. If the form of the item is not consistent with the normal dosage form of a particular controlled substance, then the presence of that controlled substance need not be eliminated unless analysis is requested by the submitting agency (i.e., the presence of LSD in white powders).

6. "No scheduled substance found" is not an acceptable finding when non-scheduled synthetic drugs are present in a sample and can be identified, either conclusively or preliminarily. The use of "no scheduled substance found" should be primarily focused on cases including, but not limited to, non-scheduled pharmaceutical type compounds (e.g. caffeine, acetaminophen, diphenhydramine, etc.), cutting agents and tableting materials (e.g. inositol, stearic acid), and samples with no compounds of interest present.

C. No Analysis Determination

1. Not all items/units need to be analyzed. Unless additional information is provided, the chemist need only identify the amount of controlled substance that would bring the highest penalty for the defendant according to the appropriate statutes. Once the highest penalty has been obtained, no further analysis will be done.

2. Factory sealed and labelled containers of cutting agents need not be analyzed (i.e., inositol, mannitol, baking soda, etc.).

3. Non-controlled pharmaceuticals with identifiable markings need not be confirmed through analysis unless there is evidence of tampering or reasons to suspect a counterfeit.

4. Factory sealed and labelled pharmaceutical preparations and blister packs do not need to be opened or confirmed through analysis unless they are scheduled substances and either the only item in a case or the item with the highest penalty.

5. In cases that are multi-unit, if the chemist observes the samples to be similar, randomly removes one that tests negative, the one tested would be reported as NSSF and the rest may be reported as No Analysis or Not Analyzed. For items not in commonly seen drug form (i.e., lima beans, marbles, rice), the one tested would be reported as NSSF and the rest may be reported as No Analysis or Not Analyzed.

MINIMUM STANDARDS AND CONTROLS FOR TESTING TECHNIQUES

Minimum standards and controls for each procedure or testing technique can be found in the relevant section of the Procedures Manual.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX IV: CHEMISTRY MINIMUM STANDARDS AND CONTROLS:

SECTION D: ADMINISTRATIVE POLICIES FOR SPECIFIC CONTROLLED SUBSTANCES

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_____

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Bureau Chief Timothy A. Tripp
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Forensic Sciences Command

APPENDIX IV

Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete an identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

ADMINISTRATIVE POLICIES FOR SPECIFIC CONTROLLED SUBSTANCES

The Illinois Controlled Substances Act governs most substances controlled in the state of Illinois. The act is found in Chapter 720, Section 570 of the Illinois Compiled Statutes. Substances controlled via administrative rule are listed separately in the Illinois Administrative Code, Title 77, Chapter X, Subchapter e, Part 2070. Additionally, the Kratom Control Act, The Methamphetamine Control and Community Protection Act, and the Methamphetamine Precursor Control Act are of interest to the Drug Chemistry Section. These can be found in Chapter 720, Sections 642, 646, and 648 respectively of the Illinois Compiled Statutes.

While the protocols, methods and procedures give directions for the analysis of suspected controlled substances, there are some substances that require specific testing in order to make a positive identification, sometimes going beyond the two-test minimum for a typical identification. These various procedures are collected into this appendix.

1. Psilocyn and Psilocybin

    a. In order to distinguish and independently identify psilocyn and/or psilocybin in a sample, one of the following methods will be employed:
        i. A Weber color test (see DC-B-1) is sufficient to indicate the presence of psilocyn, provided the following criteria are met:
            1. A primary psilocyn standard produces a positive Weber test, AND
            2. A primary psilocybin standard produces a negative Weber test, AND
            3. Both standards for (1) and (2) are tested on the same day as the sample(s), AND
            4. Both standards for (1) and (2) are logged in the casefile or a logbook (or the online equivalent). Documentation must include lot numbers or laboratory inventory number and color test results for each standard tested. Analysts may only use a verification performed by another analyst if all of the above criteria are met and the verification was logged in the logbook (or online equivalent).

                Note: If utilizing Weber reagent with a liquid extract, it must be a solvent only extract.
        ii. A prep TLC will be run indicating the presence of both psilocyn and psilocybin in a separated state, then at least one spot will be scraped and confirmed by either IR or GC/MS.
        iii. A solvent only extract will be run on TLC and if psilocyn and/or psilocybin are present in separated states, the sample will also be analyzed using GC-MS, GC-IR or IR.

b. When a negative Weber color test is produced for a sample suspected to contain hallucinogenic mushrooms, one of the other techniques listed above shall be employed to attempt to distinguish and independently identify psilocyn and/or psilocybin.
c. If the analyst cannot distinguish between psilocyn and psilocybin after attempting at least one of the techniques listed above, then the results will be reported as:

Psilocyn and/or Psilocybin. The specific compound(s) could not be differentiated.

2. Salvinorin A

a. For the analysis and storage of Salvinorin A, the following guidelines are recommended:
   i. Extract using methanol or chloroform. For leafy material, grind in mortar and pestle, shake or stir for one minute before removing the supernatant.
   ii. For small sample amounts, use boiling chloroform. Place the sample containing chloroform in a water bath; choose a setting to boil the chloroform, but not the water; evaporate the solvent to microvial volume.
   iii. Due to stability concerns, extracts should be analyzed within 24 hours.
   iv. Salvinorin A standards must be stored in the refrigerator in light-resistant or applicable containers.

3. Isomer determinations

a. Optical Isomers

   i. When Methorphan is identified in a substance that cannot be physically identified as a pharmaceutical preparation, *and* it cannot be isolated in a state sufficiently pure for effective optical isomer determination via polarimetry, then it is appropriate to identify Methorphan with a qualification that informs the agencies that the optical isomer of Methorphan could not be determined, however all of its isomers are listed in the Illinois Compiled Statutes.
   ii. When Propoxyphene is identified in a substance that cannot be physically identified as a pharmaceutical preparation, *and* it cannot be isolated in a state sufficiently pure for effective optical isomer determination via polarimetry, then it is appropriate to identify Propoxyphene with a qualification that informs the agencies that the optical isomer could not be determined.

b. Positional Isomers

   i. Synthetic cathinones, cannabinoids, opioids, and phenethylamines often have positional isomers that produce similar mass spectral data. In order to identify a specific positional isomer, analysts must have the necessary data to support the identification.
   ii. The following are options for identification of a positional isomer. (Note: not every option will work for every drug. This is dependent on the substance and instrument parameters. More than one technique may be necessary.)

1. Mass spectral comparison of the sample to the standard of the different isomers (or comparison of the mass spectra of the isomers to each other).
2. Comparison of retention time data between the sample and the standards of the different isomers.
3. Comparison of IR data obtained via FTIR or GC-IR between the sample and the standard.

iii. If a positional isomer still cannot be determined, it is acceptable to use the following remarks lines in conjunction with a general identification that does not identify the positional isomer. (The choice depends on whether or not the isomers are controlled as well.)
1. Remarks: The positional isomer of XXX could not be determined.
2. Remarks: The positional isomer of XXX could not be determined, however, all of its positional isomers are controlled in the Illinois Compiled Statutes.
   a. Example: Methylethcathinone, a synthetic cathinone.

      Remarks: The positional isomer of Methylethcathinone could not be determined, however, all of its positional isomers are controlled in the Illinois Compiled Statutes.

2. Khat

a. Suspected Khat cases containing fresh plant material should be refrigerated upon arrival at the laboratory and analyzed as soon as possible to minimize the conversion of cathinone to cathine.
b. The following technique is one possible extraction for suspected Khat:

   Grind plant material with a 1:1 mixture of cold CHCl3: Saturated Na2CO3. Sit for 10 minutes while remaining cold. Filter through glass wool, remove the chloroform layer, evaporate with air, and reconstitute in a microvial of methanol.

3. Clandestine Laboratories

i. Analytical Considerations

1. Evidence submitted from a clandestine laboratory will be limited to the identification of controlled substances and possible precursors. No capacity calculations, theoretical yields, or determination of routes of synthesis will be performed.
2. Clandestine lab samples can be solid, liquid, or a combination of the two. Liquids may be multilayered and may require sampling from each layer.
3. Testing liquids with Watesmo paper and/or pH paper can provide a good starting point for analysis. A basic aqueous sample may only require extracting with a suitable solvent. A non-aqueous sample can be injected neat but may require clean-up steps to remove interfering peaks (i.e. using hydrochloric acid fumes to precipitate out the suspected methamphetamine and then separating the precipitate from the original solvent).

4. For solids, it is best to avoid sampling any Lithium metal that may be present. Be aware that performing a Sodium Nitroprusside color test on a strongly basic item containing methamphetamine may result in a false negative red color.

ii. Safety Considerations

1. Evidence may contain strong acids or bases which can cause injuries if they come into contact with skin. Because they can also penetrate clothes or lab coats, extra care should be taken when handling clandestine lab evidence.
2. Clandestine labs often contain solvents which emit noxious or harmful vapors. For this reason, these samples should be handled in the hood, opened slowly, with the mouth of the container of evidence directed towards the rear of the hood.

iii. Storage Considerations

1. Bottles containing clandestine lab evidence should be stored upright in order to prevent leakage.
2. When possible, clandestine lab evidence should be stored in a cabinet designed for this purpose.

4. Opium

i. In order to identify Opium, the following five compounds need to be identified:
   1. Morphine
   2. Codeine
   3. Thebaine
   4. Papaverine
   5. Noscapine

ii. Analytical Considerations
   1. Preliminary
      1. Color tests: Use Mecke, Marquis or Froehde's as a preliminary test.
      2. GC/FID: Extract the sample and analyze. All five compounds' retention times must be recorded.
   2. Confirmatory
      1. GC/MS: Extract the sample and identify the above listed compounds.
      2. A primary Opium standard may also be used for comparison after the confirmation of the above compounds.

iii. It is acceptable to use the following remarks line when identifying Opium.
   1. Remarks: The five individual compounds of Morphine, Codeine, Thebaine, Papaverine and Noscapine have been identified when confirming Opium.

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ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX IV: CHEMISTRY MINIMUM STANDARDS AND CONTROLS

SECTION E: ADMINISTRATIVE POLICIES FOR FOOD PRODUCT SAMPLING

Reviewed by:

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Forensic Scientist Tiffany A. Neal, Chairperson
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Approved by:

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Bureau Chief Timothy A. Tripp
Drug Chemistry Command Coordinator
Forensic Sciences Command

APPENDIX IV

This section will provide instruction on how best to sample various food products for analysis. This will by no means be an all-inclusive list, as analysts will need to use their own discretion on a case-by-case basis.

Minimum Standards and Controls are not to be used as a substitute for good scientific procedures. On an individual case, the minimum indicated may not suffice to complete an identification as defined. It is the primary responsibility of the chemist to identify the controlled substance rather than simply fulfill these standards.

ADMINISTRATIVE POLICIES FOR FOOD PRODUCT SAMPLING

A. Guidelines

1. Food products that are consistent in appearance in a single container will be seen as one comingled unit and tested as one sample. This includes, but is not limited to, gummies (not individually wrapped), cereal, chips, cookies, and other candies.

2. Factory sealed containers that are labeled with their contents, in accordance with state guidelines, will not be analyzed unless there is evidence of tampering.

3. A large food product that has been cut, but not separated (i.e., a pan of brownies or bars), will be considered one unit and tested as one item.

4. Individually wrapped items will be treated as separate items and tested separately.

5. Because of the difficulties associated with analyzing food products due to complex matrices, only one sample from one item will initially be analyzed. The analyst will then note whether the remaining samples are consistent in appearance. Analysis on the remaining samples/items will be at the discretion of laboratory management and only completed if requested by the State's Attorney's Office.

6. If further analysis is approved to meet statutory requirements, the required number of samples must be tested individually or, if the sample allows, the submission may be analyzed using an approved sampling method. The calculated number of samples determined by the sampling method must each be individually tested to meet the Minimum Standards and Controls for analysis.

MINIMUM STANDARDS AND CONTROLS FOR TESTING TECHNIQUES

Minimum standards and controls for each individual procedure or testing technique can be found in the relevant section of the procedures manual.

ILLINOIS STATE POLICE

DRUG CHEMISTRY PROCEDURES MANUAL

APPENDIX V: MEASUREMENT UNCERTAINTY

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INTRODUCTION

All measurements are associated with a level of uncertainty. When the most significant contributing sources are taken into account, a reasonable approximation of the true uncertainty can be given. The uncertainty of measurement is a calculated range of values at a given confidence interval that allows us to express how certain we are that the "true" value is within the stated interval.

The measurement uncertainty associated with mass determination is affected by factors such as the resolution of the balance, repeatability, and linearity. Even though balances are calibrated routinely, there is still an amount of uncertainty associated with the calibration. Process uncertainty considers the reproducibility of measurement over a variety of environmental conditions, different locations, different personnel, and replicate measurements of similar objects. The standard weights utilized for function checks are calibrated at a specified interval and carry an amount of calibration uncertainty. These components are able to be quantified and used to calculate the measurement uncertainty. Once the standard uncertainties are calculated for the above contributions, the combined standard uncertainty is calculated and then used to calculate the expanded uncertainty at a confidence level of *at least 95.45%*. The expanded uncertainty is calculated for each balance readability and then utilized in the equation to calculate the total uncertainty for the mass determination.

Each individual measurement has an uncertainty associated with it. When multiple measurements are combined to obtain a total mass, their uncertainties must also combine. In order to calculate the measurement uncertainty, the root sum square (RSS) method is used.

MEASUREMENT UNCERTAINTY FOR MASS DETERMINATION

SAFETY CONSIDERATIONS

Follow the requirements of the selected procedure.

PREPARATIONS

Follow the requirements of the selected procedure.

INSTRUMENTATION

Follow the requirements of the selected procedure.

DEFINITIONS

Linearity:	The ability of the balance to have consistent sensitivity throughout the weighing range.
Process uncertainty:	This is equal to the standard deviation calculated from the function check data obtained from all personnel, laboratories, and instrumentation.
Readability:	The smallest increment of a balance's readout. For example, the Mettler Toledo XPR603 balances have a readability of 0.001 g. Also known as resolution.
Repeatability:	Taking the same measurement(s) under the same conditions.
Root Sum Square:	A statistical method of dealing with a series of values where each value is squared, the sum of these squares is calculated and the square root of that sum is then taken.
Type A uncertainty:	This type of uncertainty is estimated by statistical analysis of a series of repeated measurements.
Type B uncertainty:	This type of uncertainty is estimated by means other than from observed measurement.

MINIMUM STANDARDS AND CONTROLS

1. Calculations for the measurement uncertainty associated with mass determination must be included on the worksheet or in the case file for all reported masses greater than or equal to 0.1 gram. Measurement uncertainty does not need to be calculated for items that are not analyzed or deferred.

2. The total uncertainty must be calculated using the root sum square method. This can be completed using a LIMS worksheet included in the case file or the calculations will be shown in their entirety with the weight measurement(s) in the case notes through the matrix.
   a. The weight and uncertainty must be recorded to the same number of decimal places as the balance readability, and the uncertainty must always be rounded up (never down or truncated).

i. If more than one balance is used and one of the balances has less readability, the recorded weight and uncertainty will be recorded to the readability of the least precise balance.

3. The calculated measurement uncertainty will only be included on the report in situations where it is crucial to the determination of the weight class or penalty and could potentially result in a lower weight class.
   a. The weight and uncertainty must be reported to the same number of decimal places as the balance readability, and the uncertainty must always be rounded up (never down or truncated).
   b. Refer to the "Application of Measurement Uncertainty for Mass Determination" section below for additional reporting guidelines and truncating rules.

4. In the case that the uncertainty is deemed crucial to the determination of a weight class, analysis must continue until either enough units have been tested to negate the importance of the measurement uncertainty, or no additional units remain to be tested.

5. In cases with more than two units where an analyst will be adding together individual mass measurements to obtain a total mass, a gross weight will be recorded in the case file. In cases where it is not possible to obtain a gross weight (e.g. tablets, paper squares), a net weight of the total number of units will be obtained. Exceptions to this policy will be noted on the worksheet.

6. The measurement uncertainty evaluation budget table for each balance readability will be saved in LAM.

7. The following events will be assessed against the current budget table: addition of new personnel, acquisition of new balances, or updated calibration certificates.
   a. If there is no impact to the expanded uncertainty calculated with the MU budget table, this evaluation will be recorded in LAM.
   b. If there is a change to the expanded uncertainty, the evaluation will be recorded in LAM and the updated budget table and MU calculations will be uploaded to LAM.

PROCEDURE OR ANALYSIS

Evaluation of Measurement Uncertainty

The full evaluation of measurement uncertainty (MU) will be completed by the Command Advisory Board (CAB) chair or designee as required by QM-18. As part of the MU evaluation, the rounded expanded uncertainty will be calculated for each balance readability for Command.

If using the LIMS worksheet, the rounded expanded uncertainty will be populated for use in the calculation of the total MU.

The following items are stored in LAM: Budget tables, MU contributions, current MU value, and MU calculations.

Uncertainty Component	Distribution	Type	Divisor	Standard Uncertainty Calculation (u_s)	Component Contribution %
Linearity (*L*)	Rectangular	B	$\sqrt{3}$	$u_{s_L} = \frac{L}{(2 \times \sqrt{3})}$	$\frac{{u_s}^2}{\sum {u_s}^2} \times 100$
Resolution (*R*)	Rectangular	B	$\sqrt{3}$	$u_{s_R} = \frac{R}{(2 \times \sqrt{3})}$	
Repeatability (*r*)	Normal (k=1)	B	1	$u_{s_r} = \frac{r}{1}$	
Calibration Uncertainty (U_c)	Normal (k=2)	B	2	$u_{s_{U_c}} = \frac{U_c}{2}$	
Process Uncertainty (U_p)	Normal (k=1)	A	1	$u_{s_{U_p}} = \frac{U_p}{1}$	
Standard Weight Uncertainty (U_w)	Normal (k=2)	B	2	$u_{s_{U_w}} = \frac{U_w}{2}$	
Combined Standard Uncertainty (u_c)	$u_c = \sqrt{\left(\sum {u_s}^2\right)}$				
Expanded Uncertainty, k=2 (U)	$U = k \times u_c = 2 \times u_c$				

For each balance readability:

1. Look up the balance linearity, readability (resolution), and repeatability from manufacturer documentation for each balance type. Identify the highest value for each.

2. Evaluate process uncertainty utilizing the function check data stored since the last MU calculation.

   Determine the standard deviation for statewide data for each required weight. The process uncertainty is equal to the calculated standard deviation from the collected function check data for that date range. At least 60 total data points are needed in order to evaluate the measurement uncertainty utilizing a coverage factor of k=2.

3. Find the highest calibration uncertainty from the most recent balance calibrations at the largest weight used in the process uncertainty determination.

All balances used in casework are routinely calibrated by an ISO accredited vendor. As part of the calibration, the estimated calibration uncertainty associated with that balance is to be determined by the vendor. The uncertainty may be given as a value or a linear equation that allows the uncertainty to be calculated at any mass across the range of the balance.

4. Find the highest calibration uncertainty from the weights utilized to check the balances statewide. The standard weights utilized for function checks are calibrated as required by QM-11.

5. Enter the appropriate values into the budget table and calculations. Standard uncertainty is calculated in grams. Combined uncertainty is calculated using the root sum squares method.

6. A minimum coverage factor of k=2, corresponding to a 95.45% confidence level, will be used in determining the expanded uncertainty.

7. Update the documentation in LAM.

Events Impacting Measurement Uncertainty

In addition to the required full evaluation per QM-18, the impact of the following events on measurement uncertainty will be assessed by the Training Coordinator, CAB chair, or designee.

I. New personnel are beginning casework
II. New balances are acquired
III. Balances are recalibrated.

1. Obtain necessary data to compare to the current budget table.
    a. New personnel and balance acquisitions: A minimum of 20 data points per person and/or balance will need to be collected through the use of function check data. The data will be added to the current MU evaluation data and the standard deviation will be recalculated.
    b. Balance acquisition and calibrations: The calibration uncertainty component will be calculated with the updated calibration uncertainty equations provided by the vendor.
2. Evaluate the values from above against those in the current budget table.
    a. If the values are lower than those in the current budget table, the new information has been evaluated against the current measurement uncertainty and has been found consistent. This will be documented in LAM.

b. If the calculations show a higher standard deviation for process uncertainty or a new highest calculated calibration uncertainty, the rounded expanded uncertainty will be re-calculated.
    i. If the rounded expanded uncertainty does not change, the new information has been evaluated against the current measurement uncertainty and has been found consistent. This will be documented in LAM.
    ii. If the rounded expanded uncertainty increases, the new information is not consistent with the current measurement uncertainty and requires the budget table and calculations to be updated. This will be documented in LAM and the section will be notified of the change.

Uncertainty Calculations for Casework

Weights obtained from a single balance for a single population

The root sum square method is used to calculate the total uncertainty. In order to account for the tare of the weighing vessel with each recorded weight, the number of recorded weights will be multiplied by two. In some cases, this will be an overestimation, which is acceptable because it maintains the minimum required confidence interval.

$$U_t = \sqrt{(2N) \times (u_b)^2} = \sqrt{(2N)} \times u_b$$

U_t = total measurement uncertainty for the weighing process
N = number of recorded weights used to determine the final weight
u_b = rounded expanded uncertainty of the balance from the MU budget table

Weights obtained across multiple populations or utilizing balances with different readabilities

In the event that weights across multiple populations (case items) must be combined to reach weight class or that balances with different readabilities are utilized to obtain a weight of a single population, the total uncertainty will need to be calculated for each population and/or balance. The root sum square method is utilized for this calculation. The total uncertainty for each weighing will be rounded up to the readability of the balance prior to combining the uncertainties.

$$U_t = U_{t_{B1}} + U_{t_{B2}} + \cdots x$$

U_t = total measurement uncertainty for the sum value
$U_{t_{B1}}$ = rounded total measurement uncertainty for balance/population 1
$U_{t_{B2}}$ = rounded total measurement uncertainty for balance/population 2

Continue adding uncertainties for the number of separate balances/populations, *x*.

Application of Measurement Uncertainty for Mass Determination

1. If the total mass combined with the uncertainty could result in a change to a lower weight class, the uncertainty must be included on the report.

    a. Example 1: 1.005 g of cocaine with an uncertainty of ± 0.008 g at a 95.45% confidence level
        i. This would give a range of values between 1.013 g and 0.997 g
        ii. The value could result in a lower weight class, so the uncertainty must be included on the report.

    b. Example 2: 1.011 g (typically truncated to 1.01 g on report) of heroin with an uncertainty of ± 0.009 g at a 95.45% confidence level
        i. This would give a range of values between 1.020 g and 1.002 g
        ii. The value would remain above the weight class, so the uncertainty would not be included on the report.

    c. Example 3: 0.998 g (truncated to 0.9 g on a report) with an uncertainty of ± 0.004 g at a 95.45% confidence level
        i. This would give a range of values from 1.002 g to 0.994 g
        ii. The uncertainty would not be included on the report because the value is truncated to 0.9 g which accounts for the uncertainty of the balance in a manner which gives the benefit of the doubt to the defendant.

REPORT WORDING

When the measurement uncertainty must be included on the report, a remarks line using the following format should be used:

REMARKS: The uncertainty associated with the mass measurement in Item [Z] is [xxx] at a 95.45% confidence level.

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