## **Technical Procedure for Drug Chemistry Analysis**

- **1.0 Purpose -** This procedure specifies the required elements for the identification of controlled substances.
- **2.0** Scope This procedure applies to general casework samples in the Drug Chemistry Section at the Raleigh location of the State Crime Laboratory.

# 3.0 Definitions

- **Homogenous** Uniform.
- **Residue** An amount of material which cannot be readily removed from the container in which it was submitted.
- **4.0** Equipment, Materials and Reagents See Drug Chemistry Section technical procedures.

## 5.0 Procedure

## 5.1 Examination Documentation

- **5.1.1** The electronic FA worksheet is provided as a controlled form and shall be used as designed for casework. Forensic Scientists shall record notes which will allow another Forensic Scientist to repeat the analysis under conditions as close as possible to the original, evaluate the data, interpret the results, and form an independent conclusion.
- **5.1.2** The Drug Chemistry FA worksheet is a generic worksheet for controlled substances and clandestine laboratory casework. The comments section shall be used for explanation of tests if needed. Excel spreadsheets are an acceptable format to record and add lists of weights. These shall be imported and approved in the Case Record Object Repository for the Case Record.
- **5.1.3** The "Notes" section of the FA worksheet is provided for detailed descriptions of evidence or other information for specialized cases such as dilution/diversion.
- **5.1.4** There will be instances when plain paper is needed for note taking. Clandestine laboratory field work is one example. This is an acceptable practice as long as the notes are properly labeled, retained, and promptly scanned into the Case Record Object Repository. Any tests or analysis conducted shall include information that is included in the controlled worksheet.
- **5.1.5** Date(s) of examination shall be noted as "Date started" and "Date completed." The completion date reflects the date when all data has been incorporated into a recorded conclusion.
- **5.2** Laboratory facilities provide sufficient environmental conditions to conduct all tests included in the Section technical procedures with no further consideration required.

## 5.3 Standards and Controls

- **5.3.1** Forensic Scientists are responsible for using documented Drug Chemistry Section technical procedures outlined for the identification of controlled substances.
- **5.4 Calibrations** See Drug Chemistry Section technical procedures.

## 5.5 Application of Procedure on Evidence

# 5.5.1 Analytical Schemes

- **5.5.1.1** There are four general analytical schemes to be used for controlled substances after the physical examination of the drug form is conducted.
- **5.5.1.2** Pharmaceutical Preparation (see below for scheme).
- **5.5.1.3** Residue/Paraphernalia/Liquids (see below for scheme).
- **5.5.1.4** General Unknowns/Powders/Clandestine Tablets (see below for scheme).
- **5.5.1.5** Marijuana (see below for scheme).
- **5.5.1.6** It should be noted that sample size or other circumstances may require a rearrangement or modification of one or more steps.
- **5.5.1.7** A Forensic Scientist may encounter exhibits that require specialized analysis. For these cases the flowchart for general unknowns shall be followed and any deviations from the technical procedures shall be approved by the Drug Chemistry Technical Leader or his/her designee in accordance with the Laboratory Procedure for Authorizing Deviations.

# (ANALYTICAL SCHEMES FOLLOW)



\* Sample size or other circumstances may require rearrangement or modification of one or more steps.



\* Sample size or other circumstances may require rearrangement or modification of one or more steps.

<sup>#</sup>Refer to Drug Chemistry Section Technical Procedure for Sampling if applicable when exhibit is a liquid pharmaceutical preparation.



\* Sample size or other circumstances may require rearrangement or modification of one or more steps.



\* Sample size or other circumstances may require rearrangement or modification of one or more steps.  $^{\oplus}$  Required if macroscopic characteristics are absent or if another test is inconclusive.

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- **5.5.2** Weights (received and returned) of solids, powders, and plant material shall be recorded in the case notes. (See the Drug Chemistry Section Technical Procedure for Balances.)
  - **5.5.2.1** When it is suspected that individual units of evidence contain small amounts of heroin, the analytical balance shall be used for weight determinations.

## 5.5.3 **Reporting Weights**

- **5.5.3.1** All digits of received weights recorded in the case notes shall be reported with the associated uncertainty. See Technical Procedure for Measurement Assurance for calculations. See the Drug Chemistry Section Technical Procedure for Balances for reporting of Uncertainty of Measurement.
  - **5.5.3.1.1** Reported gross weights shall be truncated.
  - **5.5.3.1.2** Reported gross weights do not require uncertainty calculations or reporting.
- **5.5.3.2** The notation "Less than 0.1 gram" is acceptable to report recordable weights less than 0.1 gram. Uncertainty of measurement is not required when this notation is used.
- **5.5.3.3** An amount of material which cannot be readily removed from the container in which it was submitted may be reported as a residue.
- **5.5.3.4** When the sample size of an exhibit prohibits complete analysis, the reported results shall be recorded as "Insufficient sample for analysis."
- **5.5.3.5** When a sample's infrared spectrum or gas chromatogram indicates a mixture of a controlled substance(s) and non-controlled substance(s), their ratio will be evaluated based on the training and experience of the Forensic Scientist. If the overwhelming majority of the sample is indicated to be non-controlled, then the reported results shall indicate that the material contains the controlled substance(s).
  - 5.5.3.5.1 Suggested Example: Item 1: Material containing Cocaine – Schedule II. Net weight of material – 2.5 grams.
  - 5.5.3.5.2 Suggested Example: Item 1: 2.5 grams of material containing Cocaine – Schedule II.
- **5.5.3.6** The number of tablets, capsules, or other dosage units containing controlled substances shall be reported. The number returned shall be included in the case notes.

- **5.5.3.7** Liquids containing controlled substances shall measured by weights or volumes. The amount of the received liquids shall be reported. The amount of the returned liquids shall be included in the case notes.
- **5.5.4 Color tests** are used to screen evidence to determine if a controlled substance may be present. (See the Drug Chemistry Section Technical Procedure for Preliminary Color Tests.)
  - **5.5.4.1** A screening test shall be chosen based on its usefulness (i.e., microcrystalline test for cocaine, Marquis for heroin).
- **5.5.5** Ultraviolet (UV) spectroscopy may be used on extracted samples or used on straight material to screen, if it does not contain analytes that interfere with the analyte of interest. (See the Drug Chemistry Section Technical Procedure for Ultraviolet Spectroscopy.)
  - **5.5.5.1** Ultraviolet spectroscopy may be used for quantitative comparisons in cases involving dilution/diversion.
- **5.5.6 Microcrystalline tests** may be used to screen evidence or to help identify a controlled substance when used in conjunction with other technical procedures. (See the Drug Chemistry Section Technical Procedure for Polarized Light Microscopy.)
  - **5.5.6.1** When a microcrystalline test is used in conjunction with a confirmatory test (Category A), documented descriptions of the crystals shall be included in the case notes for peer review. When this method is employed, the microcrystalline test will be considered a Category C test. (See **5.5.17.**)
  - **5.5.6.2** When a microcrystalline test is used as a confirmatory test (Category B), (i.e. not in conjunction with a Category A test), the crystals shall be contemporaneously peer reviewed and a Verification Review will be entered into the case record in FA. (See **5.5.17**.)
- **5.5.7 Pharmaceutical Identifiers** Forensic Scientists shall use the markings and characteristics of pharmaceutical preparations to determine the consistency of the units and as a preliminary examination only. These identifications shall be made by using credible reference materials (e.g., *Micromedex, The Physician's Desk Reference, The Logo for Tablets and Capsules*, manufacturer's published data, and/or internet pharmacies).
- **5.5.8 Extractions/washes** Non-controlled substances, are often mixed with controlled substances and interfere with results. It may be necessary to remove them before proceeding with analysis. (See the Drug Chemistry Section Technical Procedure for Extractions and Separations.)
- **5.5.9** Infrared (IR) Spectroscopy (FT-IR) may be used to screen a sample, or it may be used to identify a controlled substance when used in conjunction with preliminary tests. (See the Drug Chemistry Section Technical Procedure for Infrared Spectroscopy.)
  - **5.5.9.1** FT-IR is used for identification when the controlled substance is not mixed with other substances, or is mixed with other substances in a ratio such that

the FT-IR spectrum of the mixture is not significantly different from that of known reference material.

- **5.5.10** Gas Chromatography Mass Spectrometry (GC-MS) may be used to screen evidence or to identify controlled substances when used in conjunction with preliminary tests. (See the Drug Chemistry Section Technical Procedure for Gas Chromatograph-Mass Spectrometry.)
  - **5.5.10.1** If the controlled substance is mixed with other substances, or in a form that is not compatible with the instrument, refer to the Drug Chemistry Section Technical Procedures for Extractions and Separations, and the Drug Chemistry Section Technical Procedure for Gas Chromatograph-Mass Spectrometry (GC-MS) for suggested sample preparation.
- **5.5.11** Gas chromatography (GC) and high performance liquid chromatography (HPLC) are used to identify controlled substances when used in conjunction with other preliminary tests. (See the Drug Chemistry Section Technical Procedure for High Performance Liquid Chromatography.)
- **5.5.12** Sampling See the Drug Chemistry Section Technical Procedure for Sampling to determine the sampling plan and population(s).
  - **5.5.12.1** There are several types of exhibits to which the sampling plan will not apply:
    - Items submitted for dilution/diversion
    - Paraphernalia
    - Partially consumed hand-rolled cigarettes
    - Young marijuana plants
    - Numerous intact marijuana plants/stalks packaged together that would be impracticable to separate
    - Residues
    - Evidence seized by the Forensic Scientist from clandestine laboratory sites
  - **5.5.12.2** The Forensic Scientist shall evaluate the evidence and submission information based on his/her training and experience, and shall determine which items will be analyzed.
  - **5.5.12.3** For each unit to be analyzed, obtain the material for analysis.
    - **5.5.12.3.1** For quantitative analysis, see the Drug Chemistry Section Technical Procedure for High Performance Liquid Chromatography and related procedures.
    - **5.5.12.3.2** For homogenous compressed materials (e.g., kilos of suspected cocaine or bricks of suspected marijuana), obtain multiple portions of material and combine for analysis.
      - Record the number of portions obtained in the case notes.

- **5.5.12.3.3** For non-homogenous compressed material, obtain multiple portions from the unit for each type of material present, and combine for analysis.
  - Record the number of portions of each material obtained in the FA case notes.
- **5.5.12.3.4** If the material is homogenous, take the amount needed for each test to be performed.
- **5.5.12.3.5** If the material is not homogenous, obtain a portion of each type of material present OR homogenize all of the material.
  - If the material was not homogenous, record in the case notes how it was homogenized.
- **5.5.12.4** If the material is a residue amount, (paraphernalia) physically remove a portion from the evidence or perform a chemical wash with a suitable solvent. The "Residue amount" option shall be used in the case notes instead of the spaces for weight received and weight returned. (See the Drug Chemistry Section Technical Procedures for Extractions and Separations for details.)
- **5.5.12.5** If the material is a liquid removed from a suspected clandestine laboratory, see the Drug Chemistry Section Technical Procedure for Clandestine Laboratories for details on collection of evidence and subsequent analysis.
- **5.5.12.6** If the material is a homogenous liquid from a regular case submission, an aliquot shall be considered a suitable portion to represent the exhibit.
- **5.5.13** Forensic Scientists shall evaluate which items to analyze in a case based on several factors. These factors include type of charge, location of item, combinations with other items resulting in elevated charges, the nature of the item (i.e., biohazard, insufficient sample, etc.).
  - **5.5.13.1** Residues shall not be analyzed if another item from the same or higher schedule has been analyzed in the case.
  - **5.5.13.2** Syringes shall be analyzed only if they are the only item in a case, or if accompanied by a written request from a prosecuting attorney. (See Drug Chemistry Section Technical Procedure for Extractions and Separations.)

#### 5.5.14 Categories of Analytical Techniques

Listed in order of decreasing discriminatory power from A to C:

Category A	Category B	Category C
Infrared Spectroscopy	Gas Chromatography	Color Tests

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Mass Spectrometry	Liquid Chromatography (HPLC)	Ultraviolet Spectroscopy
	Microcrystalline Tests	Microcrystalline Tests
	(Not used in conjunction	(Used in conjunction
	with a Category A Test)	with a Category A Test)
	Pharmaceutical Identifiers	
	Cannabis Only:	
	Macroscopic Examination	
	Microscopic Examination	
	(Counts as one each)	

- **5.5.15** When a Category A technique is incorporated into an analytical scheme, then at least one other technique (from either Category A, B, or C) shall be used.
  - **5.5.15.1** This combination must identify the specific drug(s) present.
  - **5.5.15.2** When sample size allows, the second technique shall be applied on a separate sampling.
  - **5.5.15.3** All Category A techniques shall have reviewable data.
- **5.5.16** When a Category A technique is not used, then at least three different validated techniques shall be used.
  - **5.5.16.1** This combination shall identify the specific drug(s) present and shall preclude a false positive identification. Two of the three methods shall be based on uncorrelated techniques from Category B.
  - **5.5.16.2** A minimum of two separate samplings shall be used in these three tests.
  - **5.5.16.3** All Category B techniques shall have reviewable data.
- **5.5.17** Reviewable data includes:
  - **5.5.17.1** Printed spectra and chromatograms.
  - **5.5.17.2** Reference to published data for pharmaceutical identifiers.
  - **5.5.17.3** Contemporaneous documented peer review, photographs, or digital images of microcrystalline tests if used without a Category A Test.
  - **5.5.17.4** Descriptions of microcrystalline test results, if used in conjunction with a Category A Test.
  - **5.5.17.5** For cannabis and botanical materials only: recording of detailed descriptions of morphological characteristics. (See the Drug Chemistry Section Technical Procedure for the Identification of Marijuana for descriptions used in conjunction with the FA worksheet.)

- **5.5.18** For the use of any method to be considered of value in the identification of the controlled substance, the test shall be considered positive.
  - **5.5.18.1** While negative tests provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the positive identification of a drug.
- **5.5.19** In cases where hyphenated techniques are used (e.g., GC-MS), they will be considered as separate techniques provided that the results from each are used.
- **5.5.20** Cannabis exhibits tend to have characteristics that are visually recognizable; therefore, macroscopic and microscopic examination of cannabis shall be considered as two separate Category B techniques when observations include documented botanical features as described in the Drug Chemistry Section Technical Procedure for Identification of Marijuana.
  - Additional testing shall follow the scheme outlined in Sections **5.5.15** and **5.5.16** set forth in this procedure.
- **5.5.21** For exhibits that lack sufficient observable macroscopic and microscopic botanical detail (i.e., extracts and residues), tetrahydrocannabinol (THC) shall be identified utilizing the principles in **5.5.15** and **5.5.16** set forth in this procedure.
- **5.5.22** On rare occasions, a category "A" technique may be used by itself for identification of a newly encountered analyte if data from reference material is not available. A verification review from the Technical Leader (or Forensic Scientist Manager) shall be required to document approval for these instances.
  - Data obtained from the analyte shall be compared to published reference data from a credible source recognized in the forensic community.
  - An analyte in this instance shall be defined as an unusual steroid or new designer drug.

# 5.6 Reporting

- **5.6.1** See the Drug Chemistry Section Technical Procedure for Sampling for the format to report identified substances for exhibits where sampling or sample selection has occurred.
  - **5.6.1.1** The results for identified substances from a single unit exhibit shall be reported with the name of the substance, the Schedule, and the net weight of the material.
  - **5.6.1.2** The results for non-controlled substances from a single unit exhibit shall be reported as one of the following, and the net weight of the material.
    - No controlled substances.
    - No controlled substances indicated.
    - No controlled substances identified.
  - **5.6.1.3** The results for non-controlled pharmaceutical single unit exhibits shall be reported as follows:

- The physical characteristics, including shape, color and manufacturer's markings of the unit, were visually examined and found to be consistent with a pharmaceutical preparation that does not contain a controlled substance. There were no visual indications of tampering. No chemical analysis was performed.
- **5.7 Calculations** See Drug Chemistry Section technical procedures.
- **5.8 Uncertainty of Measurement -** See the Drug Chemistry Section Technical Procedure for Measurement Assurance.
- **6.0** Limitations See Drug Chemistry Section technical procedures.
- 7.0 Safety See State Crime Laboratory Safety Manual.

# 8.0 References

ASTM Standard E2329-09. "Identification of Seized Drugs." ASTM International: West Conshohocken, PA, 2009, <u>www.astm.org</u>.

"Part III B – Methods of Analysis/Drug Identification." *Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations.* 5<sup>th</sup> Edition. January 29, 2010.

#### 9.0 Records

- FA case files
- **10.0** Attachments N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original Document – Drug Chemistry Policy 2008-DCS-01 and 2008-DCS-02 were combined and edited for conversion to ISO standards.