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 Sections 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 or to organize data. 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 necessary information not addressed in 5.1.1 or 5.1.2.

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 and administrative 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/Hashish (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)
Physical Examination of Drug Form

Pharmaceutical Preparation

Pharmaceutical Identifier

See Drug Chemistry Section Administrative Procedure for Sampling*

Non-controlled

- Single unit or obtain a single unit from the population
  - Verify non-controlled substance indicated by pharmaceutical identifier via UV/IR/GC-MS as needed (see Section technical procedures)
  - Reporting

Controlled

- Single unit or obtain a single unit from the population
  - Weigh if applicable
  - Extraction or wash if needed (see Section technical procedures)
  - MS/GC-MS and/or FT-IR
  - d,l crystals if needed
  - Reporting

* Sample size or other circumstances may require rearrangement or modification of one or more steps.
Physical Examination of Drug Form

Residue/Paraphernalia*/Liquids#

Obtain portion for analysis
(Physically remove or wash – see Section technical procedures)

Color Tests and/or Microcrystal Tests and/or UV

Extraction/wash if needed (see Section technical procedures)

MS/GC-MS and/or FT-IR

d,l crystals if needed Further Testing

Reporting Trace Evidence or other Lab section

* Sample size or other circumstances may require rearrangement or modification of one or more steps.
*Refer to Drug Chemistry Section Administrative Procedure for Sampling if applicable when exhibit is a liquid pharmaceutical preparation.
Physical Examination of Drug Form

**General Unknowns/Powders/Clandestine Tablets**

See Drug Chemistry Section Administrative Procedure for Sampling, then for each unit *

**Powders/Clandestine tablets & other unknowns**

**Weigh if applicable**

**Obtain portion for analysis**

**Color Tests and/or Microcrystal Tests and/or UV**

**Extraction/wash if needed (see Section technical procedures)**

**MS/GC-MS and/or FT-IR**

**d,l crystals if needed**

**Further Testing**

**Reporting**

**Trace Evidence or other lab section**

* Sample size or other circumstances may require rearrangement or modification of one or more steps.*
Physical Examination of Drug Form

Marijuana

See Drug Chemistry Section Administrative Procedure for Sampling, then for each unit*

Material with Marijuana Characteristics
- Record microscopic examination
- Weigh
- Obtain portion for analysis
- Record gross morphological and microscopic examinations
- Duquenois-Levine color test
- Reporting

Residues with Marijuana characteristics (smoking devices)
- Weigh or approximate volume
- Obtain portion for analysis – Physically remove or wash (see Section technical procedures)
- Duquenois-Levine color test
- GC-MS
- Reporting

Material without Marijuana characteristics (Hashish/hash oil)
- Weigh or approximate volume
- Duquenois-Levine color test
- Extraction/wash (see Section technical procedures)
- Extraction/wash if needed (see Section technical procedures)
- GC-MS
- Reporting

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

© Required if macroscopic characteristics are absent or if another test is inconclusive.
5.5.2 **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.)

5.5.2.1 A screening test shall be chosen based on its usefulness (i.e., microcrystalline test for cocaine, Marquis for heroin).

5.5.3 **Ultraviolet (UV) spectroscopy** may be used on extracted samples or used on straight material to screen. (See the Drug Chemistry Section Technical Procedure for Ultraviolet Spectroscopy.)

5.5.3.1 Ultraviolet spectroscopy may be used for cases involving dilution/diversion when known standards are submitted for comparison purposes.

5.5.4 **Microcrystalline tests** may be used to screen evidence or to help identify a controlled substance when used with other technical procedures. (See the Drug Chemistry Section Technical Procedure for Polarized Light Microscopy.)

5.5.4.1 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.

5.5.4.2 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.

5.5.5 **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.

5.5.5.1 Complete markings shall be required for identification. Tablets must be intact or complete markings must be physically matched back together if broken tablets are present.

5.5.5.2 Information obtained from partial imprints may not be used as a preliminary examination. This type of information may be included in the casefile for information purposes only.

5.5.5.3 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 such as Drugs.com and Pharmer.org).

5.5.6 **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.)
5.5.7 **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.)

5.5.7.1 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 does not interfere with a favorable comparison to the known reference material.

5.5.8 **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.)

5.5.8.1 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.

5.5.9 **Gas chromatography** (GC) may be used to identify controlled substances when used in conjunction with other preliminary tests listed in 5.5.11.

5.5.10 **Sampling** - See the Drug Chemistry Section Administrative Procedure for Sampling to determine sampling selection or sampling plan and population(s).

5.5.11 **Categories of Analytical Techniques**

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

<table>
<thead>
<tr>
<th>Category A</th>
<th>Category B</th>
<th>Category C</th>
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<tbody>
<tr>
<td>Infrared Spectroscopy</td>
<td>Gas Chromatography</td>
<td>Color Tests</td>
</tr>
<tr>
<td>Mass Spectrometry</td>
<td>Microcrystalline Tests (Not used in conjunction with a Category A Test)</td>
<td>Ultraviolet Spectroscopy</td>
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<tr>
<td></td>
<td>Pharmaceutical Identifiers</td>
<td>Microcrystalline Tests (Used in conjunction with a Category A Test)</td>
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<tr>
<td></td>
<td>Cannabis Only: Microscopic Examination Microscopic Examination (Counts as one each)</td>
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5.5.12 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.

5.5.12.1 This combination must identify the specific drug(s) present.
5.5.12.2 When sample size allows, the second technique shall be applied on a separate sampling.

5.5.12.3 All Category A techniques shall have reviewable data.

5.5.13 When a Category A technique is not used, then at least three different validated techniques shall be used.

5.5.13.1 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.

5.5.13.2 A minimum of two separate samplings shall be used in these three tests.

5.5.13.3 All Category B techniques shall have reviewable data.

5.5.14 Reviewable data includes:

5.5.14.1 Printed spectra and chromatograms.

5.5.14.2 Reference to published data for pharmaceutical identifiers.

5.5.14.3 Contemporaneous documented peer review, photographs, or digital images of microcrystalline tests if used without a Category A Test.

5.5.14.4 Descriptions of microcrystalline test results, if used in conjunction with a Category A Test.

5.5.14.5 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.)

5.5.15 For the use of any method to be considered of value in the identification of the controlled substance, the test shall be considered positive.

5.5.15.1 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.

5.5.16 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.

5.5.17 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 5.5.12 and 5.5.13 set forth in this procedure.
5.5.18 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 5.5.12 and 5.5.13 set forth in this procedure.

5.5.19 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/Forensic Scientist Supervisor) 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.

5.5.20 Weights (received and returned) of solids, powders, opiate and amphetamine tablet preparations, and plant material shall be recorded in the case notes. (See the Drug Chemistry Section Technical Procedure for Balances.)

5.5.20.1 When the analyst, based upon his/her training and experience believes that individual units of evidence contain small amounts of heroin, the analytical balance shall be used for weight determinations.

5.6 Reporting (See the Drug Chemistry Section Administrative Procedure for Sampling for the format to report identified substances for exhibits where sampling or sample selection has occurred.)

5.6.1 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 with associated uncertainty.

5.6.2 All digits of received net weights recorded in the case notes shall be reported with the associated uncertainty. See Procedure for Measurement Assurance and the Drug Chemistry Section Technical Procedure for Balances.

5.6.3 Gross weights may be recorded as needed, but shall not be reported unless sample matrix prevents complete removal of packaging. When this occurs, uncertainty of measurement does not apply to gross weights.

5.6.4 The notation “Net Weight of Material - Less than 0.1 gram” is required to report all recordable weights less than 0.1 gram when a table top balance is used to obtain the weight. Uncertainty of measurement does not apply in this situation.

5.6.4.1 When the net weight of a single pharmaceutical unit is less than 0.1 gram, it is acceptable to record an actual weight less than 0.1 gram in the casefile, and report the results as “Net Weight of (Tablet, etc.) – Less than 0.1 gram.” Uncertainty of measurement shall not be reported, even if it was calculated on an Excel spreadsheet.

5.6.4.2 When an analytical balance is used to record weights less than 0.1 gram, all digits recorded and the associated uncertainty of measurement shall be reported.

5.6.5 An amount of material which does not register on a table top balance, or an amount that cannot be readily removed from the container in which it was submitted, may be reported as a residue.
5.6.6 When a sample’s infrared spectrum indicates a mixture of a controlled substance(s) and non-controlled substance(s), the 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).

5.6.6.1 Suggested Example:
Item 1:
Material containing Cocaine – Schedule II.
Net weight of material – 2.51 (+/- 0.0X) grams.

5.6.6.2 Suggested Example:
Item 1:
2.51 (+/- 0.0X) grams of material containing Cocaine – Schedule II.

5.6.7 When the sample size of an exhibit prohibits complete analysis, the reported results shall be recorded as “Insufficient sample for analysis.”

5.6.8 The results for non-controlled substances shall be reported as “No controlled substances identified” and the net weight of the material with associated uncertainty shall be reported for the analyzed portion, if a net weight was recorded.

5.6.8.1 The results for non-controlled clandestine laboratory samples shall be reported as “No controlled substances and/or precursor chemicals identified” and the net weight of the material with associated uncertainty shall be reported for the analyzed portion.

5.6.9 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 and reported as provided in the Drug Chemistry Administrative Procedure for Sampling.

5.6.10 Liquids containing controlled substances shall be 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.

5.6.11 When a Forensic Scientist opens an item of evidence and determines it does not meet submission criteria, analysis may be terminated and the following results reported:

5.6.11.1 “The analysis on (Item # ___) has been terminated and the evidence is being returned because it does not meet Drug Chemistry Section submission guidelines.”

5.7 Calculations - See Drug Chemistry Section technical procedures.

5.8 Uncertainty of Measurement - See the Drug Chemistry Section Procedure for Measurement Assurance.

6.0 Limitations - See Drug Chemistry Section technical procedures.


8.0 References


9.0 Records

- FA case files

10.0 Attachments - N/A

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- **5.6** - “Reporting Weights” section renamed “Reporting” and moved from original Section **5.5.3**.

- **5.6.1.1** – Added reference to “associated uncertainty.”

- **5.6.1.3** – Clarification added for use with table top. (Original Section **5.5.3.2**)

- **5.6.1.5.1** and **5.6.1.5.2** – Measurement assurance data added to example. (Original Sections **5.5.3.5.1** and **5.5.3.5.2**)

5.5.7.1 - Wording changed - removed not significantly different phrase for clarification; **5.5.9** – clarified other preliminary tests as those listed in 5.5.11

5.3.1 – Added reference to section administrative procedures

5.5.17, 5.5.10, 5.6 - Revised name of Sampling Plan from Technical to Administrative Procedure

**5.5.3.1** – Removed quantitative use of UV

5.6.1.1 and **5.8** – Updated name of Procedure for Measurement Assurance

5.6.1.3 – Added “Net weight of material - “

5.6.1.5 – Removed “or gas chromatogram”, changed their ratio to the ratio

5.6.3 and **5.6.3.1** – Removed sections

Aded issuing authority to header

5.5.17 - Analytical scheme for Pharmaceutical Preparations amended to include chemical analysis to verify identity of non-controlled preparations

5.6.2 – Results for reporting of non-controlled substances changed to allow for only one option of wording, and report weight if recorded

5.5.3 - Reworded

5.5.19 – Added Forensic Scientist Supervisor

5.6.2.1 – Changed “indicated” to “identified”

5.5.5 – Added Drugs.com and Pharmer.org as examples of internet pharmacies

5.5.11 – Removed HPLC.

5.5.20 – Added weight requirement for opiate and amphetamine tablet preparations.

5.6.2 – Clarified only net weights recorded are to be reported.

5.6.3 – Clarified gross weights are only reported when sample matrix is involved.

5.6.3.1.1 – Deleted and added to 5.6.3 above.

5.6.4 – Clarified reporting of weights of pharmaceutical tablets that weigh less than 0.1 gram.

5.6.5 – Clarified use of residue amount.

5.6.8 – Clarified reporting of weights for analyzed portion of non-controlled substances.

5.6.9 – Clarified reporting of returned controlled substance tablet counts.

5.6.11 – Add wording for results when evidence does not meet submission guidelines and work is terminated.
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| **Header** - Revised issuing authority  
5.5.1.5, 5.5.1.7 – Added hashish  
5.5.5 – Reorganized section/clarified requirement for all markings to be present on pharmaceuticals if database identification is used as a preliminary examination  
5.5.5.2 - Partial imprint information may be included in the casefile, but not used as a preliminary examination |