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

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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 Analytical Techniques

5.5.1 The following analytical techniques are validated for use. See **5.8** for identification criteria.

5.5.1.1 Color tests - See the Drug Chemistry Section Technical Procedure for Preliminary Color Tests.

5.5.1.2 Microcrystalline tests - See the Drug Chemistry Section Technical Procedure for Polarized Light Microscopy.

5.5.1.2.1 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.

5.5.1.2.2 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.

5.5.1.3 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.

5.5.1.3.1 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.

5.5.1.3.2 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.

5.5.1.3.3 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).

5.5.1.3.3.1. If a software program reference is used, the version number shall be listed in the casefile.

5.5.1.4 Extractions/washes - See the Drug Chemistry Section Technical Procedure for Extractions and Separations.

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5.5.1.5 Infrared (IR) Spectroscopy (FTIR) - See the Drug Chemistry Section Technical Procedure for Infrared Spectroscopy.

5.5.1.5.1 FTIR 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 FTIR spectrum of the mixture does not interfere with comparison to the known reference material.

5.5.1.6 Gas Chromatography (GC) or Gas Chromatography-Mass Spectrometry (GC-MS) - See the Drug Chemistry Section Technical Procedure for Gas Chromatograph/Mass Spectrometry (GC-MS).

5.5.1.6.1 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.

5.6 Analytical Schemes

5.6.1 There are general analytical schemes to be used for analysis of suspected controlled substances. See the **Drug Chemistry Analytical Schemes Working Instructions** form. These schemes are not intended to include evidence from submitted clandestine laboratories. These items shall be processed according to the Technical Procedure for Clandestine Laboratory Analysis.

5.6.2 After the physical examination of the drug form is conducted, the Drug Chemistry Section Administrative Procedure for Sampling shall be consulted to determine the sample selection method or if sampling applies to the population being analyzed.

5.6.3 It should be noted that sample size or other circumstances may require a rearrangement or modification of one or more steps.

5.6.4 A Forensic Scientist may encounter exhibits that require specialized analysis. For these cases any deviations from the technical procedures or work instructions shall be approved by the Drug Chemistry Technical Leader or designee in accordance with the Laboratory Procedure for Authorizing Deviations.

5.6.5 The chosen analytical scheme shall demonstrate the identity of the specific drug present and shall preclude a false positive identification and minimize false negatives. Where a scheme has limitations, this shall be reflected in the final interpretation.

5.7 Categories of Analytical Techniques

Listed in order of decreasing discriminatory power from A to C and specified for the techniques currently utilized at the North Carolina State Crime Laboratory:

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Category A	Category B	Category C
Infrared Spectroscopy	Gas Chromatography	Color Tests
Mass Spectrometry	Microcrystalline Tests (Not used in conjunction with a Category A Test)	Microcrystalline Tests (Used in conjunction with a Category A Test)
	Cannabis Only: Macroscopic Examination Microscopic Examination (Counts as one each)	Pharmaceutical Identifiers

5.8 Criteria for Identification

5.8.1 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.

5.8.1.1 This combination must identify the specific drug(s) present.

5.8.1.2 When sample size allows, the second technique shall be applied on a separate sampling.

5.8.1.3 All Category A techniques shall have reviewable data.

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

5.8.2.1 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.

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

5.8.2.3 All Category B techniques shall have reviewable data.

5.8.3 Reviewable data includes:

5.8.3.1 Printed spectra and chromatograms.

5.8.3.2 Reference to published data for pharmaceutical identifiers.

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

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

5.8.3.5 For cannabis and botanical materials only: recording of detailed descriptions of morphological characteristics. (See the Drug Chemistry Section Technical Procedure for the Identification of Plant Material and Plant Material Extracts for descriptions used in conjunction with the worksheet.)

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

5.8.4.1 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.

5.8.5 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.

5.8.6 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 Plant Material and Plant Material Extracts.

- Additional testing shall follow the scheme outlined in Section **5.6** and set forth in this procedure.

5.8.7 Only reference materials with authenticating documentation added to in-house generated reference collections may be used to identify controlled substances.

5.8.7.1 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 designee, 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. The reference data shall be included in the case file.

- An analyte in this instance shall be defined as an unusual steroid, a new designer drug, or an isomer of an existing drug.

5.8.8 **Weights** - All digits of received net weights recorded in the case notes for analyzed item(s) shall be reported with the associated uncertainty and a statement of the level of confidence, with the exception of pharmaceutical delivery systems and items worked under the Hypergeometric Sampling plan. See Procedure for Measurement Assurance and the Drug Chemistry Section Technical Procedure for Balances.

5.8.8.1 For item(s) not analyzed in a case, but for which a net weight is recorded, the net weight shall not be reported.

5.8.8.2 Should an additional analysis be requested for an item in which a net weight was previously recorded, the original net weight shall be reported. A notation shall be made in the worksheet that the reported weight was taken from the previous examination/casefile.

5.8.8.3 The analytical balance may be used for weight determinations of small amounts of material (e.g., bindles, tablets, etc.) when hypergeometric sampling or a sample selection method applies to the item.

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5.8.8.4 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.

5.8.8.5 An amount of material that cannot be readily removed from the container in which it was submitted, shall be reported as a residue.

5.8.8.6 If material can be removed from the container in which it was submitted, but the amount of material does not register on the tabletop balance, it shall be reported as "Less than 0.01 gram".

5.8.8.7 Net weights recorded less than the associated uncertainty shall be reported without the uncertainty of measurement.

5.9 Reporting - See **Appendix A** for the format to report identified substances for exhibits where sampling or sample selection has occurred.

5.9.1 The results for identified substances shall be reported with the name of the substance and the net weight of the material with associated uncertainty. See **5.8.8** for weight exceptions.

5.9.1.1 Substances not specifically listed in statute shall first be evaluated to determine if it is a controlled substance, a potential controlled substance analog, a controlled substance isomer, and/or a byproduct or degradant of a controlled substance.

5.9.1.1.1 An identified substance found to be controlled shall be reported in accordance with **5.9.1**.

5.9.1.1.2 A potential controlled substance analog, as defined by statute, shall first be evaluated to determine if it is substantially similar to a Schedule I or Schedule II controlled substance.

A substance will be considered substantially similar if any of the following are satisfied:

- The substance differs in no more than two (2) atoms, one (1) functional group, or any combination thereof, from the structure of a controlled substance. A functional group being that of an alkyl, alkene, alkyne, arene, haloalkane, haloalkyne, haloalkene, aromatic halide, alcohol, ether, amine, aldehyde, ketone, carboxylic acid, ester, or amide group;
- The substance: (1) differs in its chemical structure to a controlled substance only by substituting one or more hydrogens with halogens or by substituting one halogen with a different halogen; or (2) is an alkyl homolog of a controlled substance; or

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- The substance in question shares a common core structure (the central portion of the molecule is the same) with a controlled substance in schedules I or II and has only one point of divergence from the controlled substance.

5.9.1.1.3 After evaluation, a substance deemed substantially similar shall be reported in accordance with **5.9.4** unle**ss** it is the only substance identified in the case, in which case it shall be reported in accordance with **5.9.1.** A note may be made in the worksheet indicating "X, the chemical structure of which is substantially similar to the chemical structure of Y, a Schedule Z controlled substance," where X is the identified substance; Y is the substance currently listed in statute; and Z corresponds to the current Schedule listed in statute for substance Y.

5.9.1.1.4 The evaluation process will determine "Y" and "Z" in **5.9.1.1.3**, and shall be documented by the Drug Chemistry Section Technical Leader or his/her designee.

5.9.1.1.5 After evaluation, an identified substance may be an isomer of a controlled substance and as applicable, shall be reported in accordance with **5.9.1**. In addition, the substance may be found to be a potential analog, as determined by the criteria in **5.9.1.1.2**. Substances found to satisfy **5.9.1.1.2** shall be reported in accordance with **5.9.1.1.3.** Substances that do not satisfy **5.9.1.1.2** shall be handled according to **5.9.1.2.**

5.9.1.1.6 After evaluation, an identified substance determined to be a byproduct, or degradant of a controlled substance, shall be reported in accordance with **5.9.4** unless it is the only substance in the case, in which it shall be reported in accordance with **5.9.1**.

5.9.1.2 A substance that is evaluated and is not found to be a controlled substance or does not fall under **5.9.1.1.2**, **5.9.1.1.5, or 5.9.1.1.6** shall be reported in accordance with **5.9.4.** An exception may be made by the Drug Chemistry Section Technical Leader or his/her designee on a case by case basis.

5.9.2 Reporting statements not included in **Appendix A** may be needed to convey the analysis results. These reporting statements shall be approved by the Drug Chemistry Technical Leader, or designee, and shall be documented in the case record.

5.9.3 The "TYPE EXAMINATION REQUESTED" for submitted clandestine laboratories shall be listed as "Examine for controlled substances" when there is no additional request from the customer.

5.9.3.1 When a specific request for analysis of pseudoephedrine (or ephedrine) (i.e. precursors) is made by the customer in these type cases, the "TYPE EXAMINATION REQUESTED" shall be listed as "Examine for controlled substances and/or (pseudo)ephedrine."

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5.9.4 The results for non-controlled substances shall be reported as "No controlled substances identified" with the appropriate statutory reference (see **Appendix A**) and the net weight of the material with associated uncertainty shall be reported for the analyzed portion, if a net weight was recorded.

5.9.4.1 When no specific additional analysis is requested by the customer, the results for non-controlled clandestine laboratory samples shall be reported as "No controlled substances identified" (with the appropriate statutory reference, see **Appendix A**) and the net weight of the material with associated uncertainty shall be reported for the analyzed portion.

5.9.4.2 When a specific request for analysis of (pseudo)ephedrine (i.e. precursors) is made by the customer, and analysis fails to identify a controlled substance or (pseudo)ephedrine in clandestine laboratory samples, the results shall be reported as "No controlled substances or (pseudo)ephedrine identified" (with the appropriate statutory reference, see **Appendix A**). The net weight of the material with associated uncertainty shall be reported for the analyzed portion.

5.9.5 The number of tablets, capsules, or other dosage units containing controlled substances shall be recorded in the casefile and reported as provided in **Appendix A**. The number analyzed shall be included in the case notes.

5.9.6 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.

5.9.7 When a Forensic Scientist opens a case and determines all of the item(s) of evidence do not meet submission criteria, no analysis shall be performed and the following results reported:

5.9.7.1 "The analysis on this case has been terminated and the evidence is being returned because it does not meet Drug Chemistry Section submission guidelines."

5.9.8 If the analytical data produced is insufficient for the identification of any compound in an item, the item shall be reported as "No Controlled Substances Identified" (with the appropriate statutory reference, see **Appendix A)**.

5.10 Calculations - See Drug Chemistry Section technical procedures.

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

6.0 Limitations - See Drug Chemistry Section technical procedures.

7.0 Safety - See State Crime Laboratory Safety Manual.

7.1 Cases submitted to the drug chemistry section have a possibility of being harmful to the analyst.

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7.2 All scientists shall wear at minimum gloves and a lab coat when working with open evidence and/or chemicals/reagents. In addition, protective eyewear is highly recommended. Particle masks (e.g. N95 dust masks) may be used for the comfort of the analyst.

7.3 Scientists should utilize a "buddy-system," notifying at least one other individual they are working in the laboratory.

7.4 Once the case is analyzed and it has been determined that fentanyl or a fentanyl analogue is contained in the evidence, the scientist shall ensure the evidence is double bagged for return to the agency.

8.0 References

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

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

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations. 8th Edition. June 13, 2019.

National Alliance for Model State Drug Laws. Model Controlled Substances Analogue Act. Harrisburg, PA, 2018. (Model Controlled Substance Analogue Act (namsdl.org)).

9.0 Records

- FA case files
- Drug Chemistry Analytical Scheme Working Instructions

10.0 Attachments –
Appendix A – Reporting Templates

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Revision History		
Effective Date	Version Number	Reason
05/03/2024	15	**5.6.1 –** Clarified that analytical schemes do not apply to clandestine lab evidence **5.8.7** – Added requirements for in-house reference collections Previous **5.8.7** – New **5.8.7.1**: Indented in/covered under **5.8.7** **5.8.8.5** – Previous **5.9.3**: Consolidated under weights **5.8.8.6** – Previous **5.9.4**: Consolidated under weights **5.8.8.7** – Added **5.9.1.1** – Updated to include additional compound types that need to be evaluated **5.9.1.1.3** – Updated reference to previous paragraphs **5.9.1.1.5** - Indented old **5.9.1.2** **5.9.1.1.6** – Added reporting requirements for byproducts, and/or degradants New **5.9.1.2 –** Old **5.9.1.3**: Updated references to previous paragraphs **7.2 –** Clarified when PPE needs to be worn in the lab

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Appendix A – Reporting Templates

(SAMPLE SELECTION - PHARMACEUTICAL / CONTROLLED)

One tablet (or other unit description as needed) was analyzed and found to contain
Identified Substance(s).
Net weight of tablet (unit) - X.XX (+/- X.XX) gram(s).

X tablet(s) (units) was/were visually examined; however no chemical analysis was performed.
Net weight of tablet(s) (units) - X.XX (+/- X.XX) gram(s).

The physical characteristics, including shape, color, and manufacturer's markings of all units were visually examined and found to be consistent with a pharmaceutical preparation containing Identified Substance(s).
There were no visual indications of tampering.

(SAMPLE SELECTION - PHARMACEUTICAL / NON-CONTROLLED)

One tablet (or other unit description as needed) was analyzed and found to contain
No Controlled Substances Identified in accordance with North Carolina General Statute Chapter 90 **or**
No Controlled Substances Identified in accordance with Title 21 United States Code (USC) Controlled Substances Act
Net weight of tablet (unit) – X.XX (+/- X.XX) gram(s).

X tablet(s) was/were visually examined; however no chemical analysis was performed.
Net weight of tablet(s) (units) - X.XX (+/- X.XX) gram(s).

The physical characteristics, including shape, color, and manufacturer's markings of all units were visually examined and found to be consistent with a pharmaceutical preparation containing No Controlled Substances Identified. There were no visual indications of tampering

(SAMPLE SELECTION - NON-PHARMACEUTICAL / CONTROLLED)

X (*units)* (was)/(were individually) analyzed and (was)/(were each) found to contain
Identified Substance(s).
Net weight of material - X.XX (+/- X.XX) gram(s).

X *(units)* - No Chemical Analysis.

(SAMPLE SELECTION - NON-PHARMACEUTICAL / NON-CONTROLLED)

X (*units)* (was)/(were individually) analyzed and (was)/(were each) found to contain
No Controlled Substances Identified in accordance with North Carolina General Statute Chapter 90 **or**
No Controlled Substances Identified in accordance with Title 21 United States Code (USC) Controlled Substances Act
Net weight of material - X.XX (+/- X.XX) gram(s).

X *(units)* - No Chemical Analysis.

(HYPERGEOMETRIC – UNITS IN THE POPULATION CONTAIN PACKAGING, EX: BINDLES/BAGGIES)

Item X was analyzed with a hypergeometric sampling plan that demonstrates with 95% confidence that at least 90% of the packaged items (e.g. bindles, baggies) contain the identified substance(s) as listed below.

X (units) were individually analyzed and were each found to contain:
Identified compound(s).
Net weight of X units – X.XX/X.XXXX (+/- X.XX/X.XXXX) gram(s).

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X units (the remainder of the 90%) were not individually analyzed nor weighed.

An estimated net weight for the material contained within the X units (the entire 90% of item X) was calculated to be X.XX/X.XXXX (+/- X.XX/X.XXXX) gram(s).

The remaining approximate 10% of item X consists of X units which were not chemically analyzed nor included in the extrapolated weight.

FOR NON-HOMOGENOUS RESULTS ONLY:
XX of the XX analyzed (units) also contained Identified compound(s).

(HYPERGEOMETRIC – UNITS IN THE POPULATION DO NOT CONTAIN PACKAGING, EX: TABLETS)

Item X was analyzed with a hypergeometric sampling plan that demonstrates with 95% confidence that at least 90% of the submitted items (e.g. tablets) contain the identified substance(s) as listed below.

X (units) were individually analyzed and were each found to contain:
Identified compound(s).
Net weight of X units – X.XX/X.XXXX (+/-X.XX/X.XXXX) gram(s).

X units (the remainder of the 90%) were not individually analyzed but a single combined net weight was obtained. The net weight of the X units is X.XX/X.XXXX (+/-X.XX/X.XXXX) gram(s).

The remaining approximate 10% of item X consists of X units which were not chemically analyzed nor weighed.

FOR NON-HOMOGENOUS RESULTS ONLY:
XX of the XX analyzed (units) also contained Identified compound(s).

**These reporting templates are to be used for populations where the analyzed units are found to contain identical results, where applicable, unless otherwise designated.*

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