PART III B

METHODS OF ANALYSIS/ANALYTICAL SCHEME FOR IDENTIFICATION OF DRUGS OR CHEMICALS

The purpose of PART III B is to recommend minimum requirements for the forensic identification of seized drugs or chemicals. A reliable and scientifically supported identification of a drug or chemical depends on the use of an appropriate analytical scheme by competent analysts in a quality-controlled process. PART III B addresses the overall selection of techniques upon which validated methods and procedures are applied in the laboratory.

IIIB.1 Introduction

An analytical scheme is a combination of selected techniques used to reach a scientifically supported conclusion. For example, the scheme required to answer the question “is methamphetamine present?” would be different from the scheme needed to answer the question “is the isomer l-methamphetamine present?”

IIIB.2 Elements of an Analytical Scheme

IIIB.2.1 Techniques incorporated within the analytical scheme can be classified into three categories based upon the level of selectivity they achieve (Figure 1).

IIIB.2.2 An appropriate analytical scheme shall achieve a sufficient level of selectivity to enable a scientifically supported conclusion relevant to the jurisdiction.

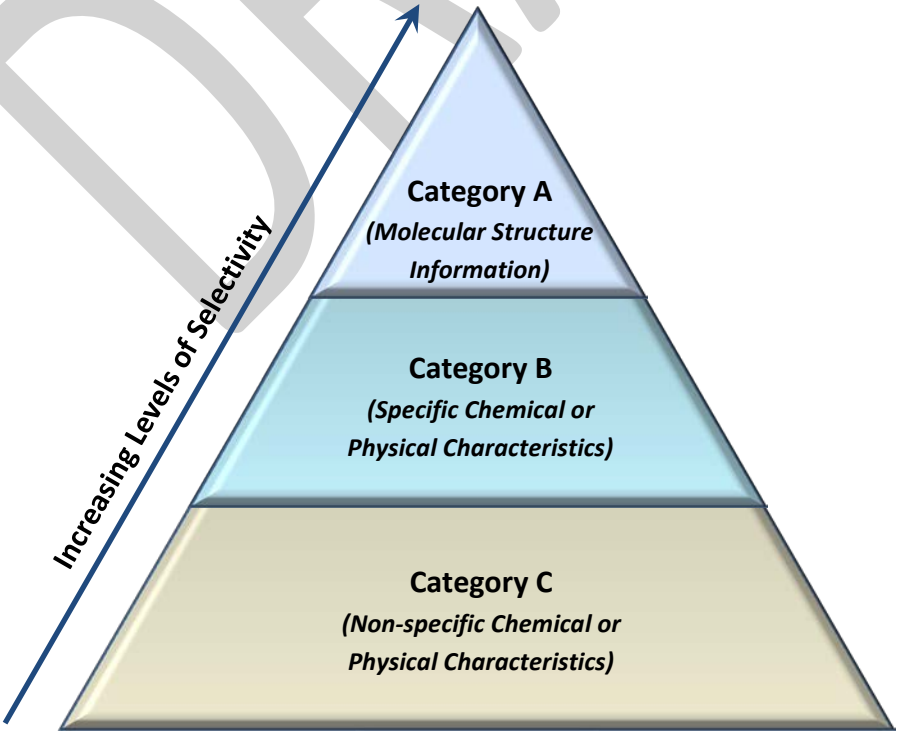

Figure 1 – Levels of Selectivity

IIIB.2.3 Table 1 lists techniques that can be incorporated within an analytical scheme for the identification of drug(s) or chemical(s). Techniques are listed according to the potential level of selectivity. Category A techniques provide the highest level of selectivity through structural information, Category B techniques provide an intermediate level of selectivity through physical / chemical characteristics, and Category C techniques achieve a low level of selectivity but provide corroborative information.

Category A *(Highest Selectivity)*	Infrared Spectroscopy
	Mass Spectrometry
	Nuclear Magnetic Resonance Spectroscopy
	Raman Spectroscopy
	X-ray Diffractometry[1]
Category B *(Intermediate Selectivity)*	Capillary Electrophoresis
	Gas Chromatography
	Ion Mobility Spectrometry
	Liquid Chromatography
	Microcrystalline Tests
	Supercritical Fluid Chromatography
	Thin Layer Chromatography
	Ultraviolet/Visible Spectroscopy (full spectrum)
	Macroscopic Examination (Cannabis only)
	Microscopic Examination (Cannabis only)
Category C *(Lowest Selectivity)*	Color Tests
	Fluorescence Spectroscopy
	Immunoassay
	Melting Point
	Pharmaceutical Identifiers[2]

Table 1 – Categories of Analytical Techniques[3]

[1] Data obtained from single crystal samples (X-Ray Crystallography) have the potential to be classified as Category A. However, diffraction data collected from powders are generally limited to Category B.

[2] Pharmaceutical Identifiers may provide a high degree of selectivity, but due to the potential for counterfeits, the technique has been placed in Category C.

IIIB.2.4 Since the identification of a drug or chemical can be achieved using a variety of techniques in different combinations, the analysts must design their analytical scheme based on suitable techniques and the requirements of their jurisdiction. (see SD-7 for examples).

IIIB.2.5 When building an analytical scheme, the laboratory shall include appropriate quality practices (see PART IVA). Relevant limitations of the scheme shall be documented and reported, as required. (see IVA PART IVC.2.2).

IIIB.2.6 It is the responsibility of the laboratory's management to provide adequate instrumentation / equipment to allow the appropriate combination of analytical techniques that achieves identification and meets the requirements of its jurisdiction.

IIIB.3 Minimum Requirements for an Analytical Scheme for Drugs and Chemicals

IIIB.3.1 When a Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used to support the identification.

IIIB.3.1.1 A technique is considered Category A when the spectral data obtained provide structural information, a high level of selectivity, and are reviewable.

IIIB.3.1.2 A Category A technique may not provide sufficient selectivity when:

IIIB.3.1.2.1 The mode of operation or the level of resolution of the technique limits the ability to distinguish the analyte from structurally similar or related compounds;

IIIB.3.1.2.2 The properties or complexity of the sample limit the ability to distinguish the analyte of interest; or

IIIB.3.1.2.3 The quantity of the sample or concentration of the analyte is limited.

IIIB.3.1.3 In circumstances where limitations are observed, the technique may still form part of an analytical scheme provided the limitations are addressed through the use of another suitable technique within the scheme, which

[3] Techniques within categories are listed in no particular order or ranking.

achieves the overall level of selectivity required for identification.

IIIB.3.2 When a Category A technique is not used, at least three separate techniques shall be employed; two shall be from Category B, the combination of which provides a high degree of selectivity. The third technique (either Category B or C) is required to support the identification.

IIIB.3.2.1 A high degree of selectivity is achieved when the two chosen Category B techniques operate on different analytical principles (e.g., GC and LC) or exploit different chemical and physical properties of the analytes (e.g., GC using two different stationary phases which achieve different interactions with the analyte).

IIIB.3.3 For the results of the techniques within the analytical scheme to be considered of value towards the identification of the analyte, the test results must be positive, meet all quality control requirements, and achieve the selectivity required of its category.

IIIB.3.3.1 A result is considered positive when it fulfils the laboratory-defined acceptance criteria for the test. Tests shall include comparisons using a suitable reference material or external reference data, or include structural elucidation (see PART IVA.6.1.6).

IIIB.3.3.2 While negative results may provide useful information, these results do not contribute towards establishing the identification of the drug or chemical of interest.

IIIB.3.3.3 When the test result does not achieve the level of selectivity of its category, additional technique(s) may be required within the analytical scheme.

IIIB.3.4 A hyphenated technique (e.g. gas chromatography-mass spectrometry, liquid chromatography-ultraviolet/visible spectroscopy) may be considered as two separate techniques within the analytical scheme provided the criteria for positive results are fulfilled for both techniques.

IIIB.3.5 The analytical scheme provides a scientifically supported conclusion when each technique achieves the level of selectivity required of its category and the positive test results corroborate each other.

IIIB.3.6 Relevant limitations of the scheme shall be documented and reported, as required.

IIIB.4 Minimum Requirements for an Analytical Scheme for Botanicals

IIIB.4.1 For herbal cannabis, macroscopic and microscopic examinations will be considered as different techniques from Category B when observations include documented details of botanical features. Laboratories shall define the acceptance criteria for these botanical features for each examination.

IIIB.4.2 For exhibits of cannabis that lack sufficient observable macroscopic and microscopic botanical detail (e.g., extracts or residues), Δ9-tetrahydrocannabinol (THC) or other cannabinoids shall be identified utilizing the principles described above (PART IIIB.3) for building an analytical scheme.

IIIB.4.3 Alternatively, cannabis and other botanical material may be identified by botanists utilizing morphological characteristics alone provided sufficient botanical features appropriate for identification are observed and documented. Such examinations shall be made only by analysts competent in botanical identifications. In this context, competency exclusively applies to those examiners recognized as professional botanists or those who are appropriately trained in botanical identification.

IIIB.4.4 Identifications of chemical components contained in botanicals (e.g., mescaline, opiates, psilocin) shall rely on principles described above (PART IIIB.3) for building an analytical scheme.

IIIB.5 Quality Practices

IIIB.5.1 All Category A and B techniques shall have data that are reviewable. Examples of reviewable data for various techniques include:

- instrumental techniques – spectra, chromatograms, or images
- TLC – images, photocopies (color, when possible) or contemporaneous documented peer-reviewed notes
- microcrystalline tests – images or contemporaneous documented peer-reviewed notes
- cannabis and botanical materials – contemporaneous detailed descriptions of morphological characteristics

IIIB.5.2 The laboratory shall employ quality practices to ensure the results correspond to the sample tested. Examples include:

- removing two aliquots from the sample and testing them independently
- using sample identification procedures such as bar-coding and witness checks
- using good laboratory practices (e.g., positive and negative controls, one sample opened at a time, procedural blanks)