SUPPLEMENTAL DOCUMENT SD-7
For Part III B - Methods of Analysis/Analytical Scheme for Identification of Drugs or Chemicals

Construction of an Analytical Scheme

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Purpose
The purpose of this supplemental document is to provide guidance on the construction of appropriate analytical schemes as required by SWGDRUG Recommendations Part III B and III C.

Definition
An ***analytical scheme*** is a combination of selected techniques used to reach a scientifically supported conclusion.

Introduction
The minimum requirements for constructing an analytical scheme are put forth in Part III B *Methods of Analysis/Analytical Scheme for Identification of Drugs or Chemicals*. For convenience of the reader, the Levels of Selectivity (Figure 1) and Categories of Analytical Techniques (Table 1) are included here as a reference.

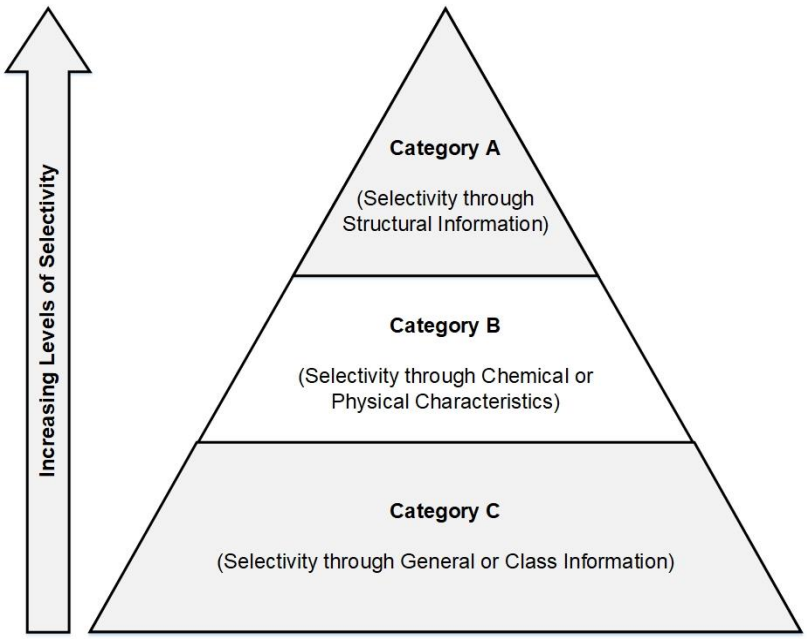

Figure 1 – Levels of Selectivity

Table 1 – Categories of Analytical Techniques[1]

Category A (Selectivity through Structural Information)	Infrared Spectroscopy
	Mass Spectrometry
	Nuclear Magnetic Resonance Spectroscopy
	Raman Spectroscopy
	X-ray Diffractometry[2]
Category B (Selectivity through Chemical and Physical Characteristics)	Capillary Electrophoresis
	Gas Chromatography
	Ion Mobility Spectrometry
	Liquid Chromatography
	Microcrystalline Tests
	Supercritical Fluid Chromatography
	Thin Layer Chromatography
	Ultraviolet/Visible Spectroscopy[3]
	Macroscopic Examination (Cannabis only)
	Microscopic Examination (Cannabis only)

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

[2] X-ray Diffractometry provides crystallographic structural information, rather than molecular structural information.

[3] Ultraviolet/Visible Spectroscopy, when used with a wavelength range, has been placed in Category B.

Category C (Selectivity through General or Class Information)	Color Tests
	Fluorescence Spectroscopy
	Immunoassay
	Melting Point
	Pharmaceutical Identifiers[4]

When constructing an analytical scheme, the achieved selectivity of the technique in the context of the particular analysis must be considered. Where any selected technique does not achieve the intended level of selectivity, then the analytical scheme may require additional techniques in order to provide a scientifically supported conclusion.

These example analytical schemes are not intended to be all inclusive. Each of these analytical questions has multiple options for analytical schemes that would achieve a correct answer. These examples were selected to demonstrate different ways techniques can be used within a particular scheme. A discussion of considerations follows each example.

Note: Throughout the following examples, it is assumed that the laboratory is utilizing validated methods and employing quality practices to ensure the results correspond to the sample tested. Examples of these practices include:

- *removing two aliquots from the sample and testing them independently;*
- *employing 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).*

In schemes where solvents are specified it is assumed that the extractions performed by the analyst are appropriate for both the sample matrix and the detection of compounds of interest to the laboratory.

Examples of Selected Schemes for Analysis of Seized Drugs

Question #1: Does the sample contain heroin?
Scheme Selected #1: GC-MS (Category B + A)

Category	Technique	Result	Assessment
B	GC	Retention time (t_R) of analyte peak is consistent with heroin reference material	Information provides Category B selectivity and the result is consistent with heroin

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

A	MS (EI)	Spectrum of analyte is consistent with heroin reference material	Information provides Category A selectivity and the result is consistent with heroin

Discussion: Each technique achieves the level of selectivity required and the positive test results corroborate each other. The scheme of GC (Category B) and MS (Category A) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains heroin.

Question #2: Does the sample contain either ephedrine or pseudoephedrine?
Scheme Selected #2: GC-MS (Category B + A)

Category	Technique	Result	Assessment
B	GC	t_R of analyte peak is consistent with pseudoephedrine reference material	Information provides Category B selectivity and the result is consistent with ephedrine, pseudoephedrine, or a mixture of both
A	MS (EI)	Spectrum of analyte is consistent with pseudoephedrine reference material	Information provides Category A selectivity and the result is consistent with ephedrine, pseudoephedrine, or a mixture of both

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other. Even though the scheme does not allow the discrimination between ephedrine and pseudoephedrine, the data from GC (Category B) and MS (Category A) provide a scientifically supported conclusion to the question asked and, therefore, the scheme is fit for purpose.

Conclusion: The sample contains ephedrine/pseudoephedrine.

Question #3: Does the sample contain ephedrine?
Note the difference from Question 2 above: now, ephedrine specifically has to be identified, rather than identification of ephedrine/pseudoephedrine.
Scheme Selected #3: GC-MS (Category B + A)

Category	Technique	Result	Assessment
B	GC	t_R of analyte peak is consistent with ephedrine reference material but is indistinguishable from pseudoephedrine reference material	Information provides Category B selectivity and the result is consistent with ephedrine, pseudoephedrine, or a mixture of both, but does not provide sufficient separation to answer the question; another technique is required

A	MS (EI)	Spectrum of analyte is consistent with ephedrine reference material. Structural information is provided, but it is indistinguishable from pseudoephedrine reference material	Information provides Category A selectivity and the result is consistent with ephedrine, pseudoephedrine, or a mixture of both
Additional Technique: LC (Category B)			
B	LC	t_R of analyte peak is consistent with ephedrine reference material and distinguishable from pseudoephedrine reference material	Information provides Category B selectivity and the result is consistent with ephedrine

Discussion: The selected scheme of GC-MS (Category B + Category A) was sufficient to identify ephedrine/pseudoephedrine, but did not specifically identify ephedrine as required. Although the mass spectrum provided structural information, the information was insufficient to differentiate between stereoisomers. Another technique (LC - Category B) was necessary to obtain the selectivity to differentiate the two compounds in question.

The enhanced scheme (with inclusion of the additional technique) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains ephedrine.

Question #4: Does the crystalline sample contain methamphetamine?
Scheme Selected #4: ATR-FTIR (Category A) + Color test (Category C)

Category	Technique	Result	Assessment
A	ATR-FTIR	Spectrum consistent with methamphetamine HCl reference material	Information provides Category A selectivity and the result is consistent with methamphetamine HCl
C	Color Test	Positive color change consistent with methamphetamine HCl reference material	Information provides Category C selectivity and the result indicates methamphetamine or related compounds

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other. The scheme of ATR-FTIR (Category A) and color test (Category C) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose. In addition, the ATR-FTIR provided salt form information, which was not part of the question.

Conclusion: The sample contains methamphetamine.

Question #5: Does the powder sample contain methamphetamine?
Scheme Selected #5: ATR-FTIR (Category A) + Color test (Category C)

Category	Technique	Result	Assessment
A	ATR-FTIR	Mixed spectrum with few significant peaks attributable to methamphetamine	Information does not provide Category A selectivity since there are insufficient identification features; another technique is required
C	Color Test	Positive color change consistent with methamphetamine HCl reference material	Information provides Category C selectivity and the result indicates methamphetamine or related compounds
Additional Technique(s): GC-MS (Category B + A)			
B	GC	t_R of analyte peak is consistent with the methamphetamine HCl reference material, but inconsistent with the phentermine reference material	Information provides Category B selectivity and the result is consistent with methamphetamine
A	MS (EI)	Spectrum of analyte is consistent with methamphetamine HCl reference material	Information provides Category A selectivity and the result is consistent with methamphetamine

Discussion: The selected scheme of the ATR-FTIR (Category A) and Color Test (Category C) was insufficient to identify methamphetamine within the mixture, but did provide information on the class of compounds (an amphetamine). The ATR-FTIR did not provide suitable structural information, so another technique (MS - Category A) was chosen. In addition, the hyphenated GC-MS test provided retention time information to further support the conclusion.

The enhanced scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains methamphetamine.

Question #6: Does the powder sample contain cocaine?
Scheme Selected #6: Raman (Category A) + Color test (Category C)

Category	Technique	Result	Assessment
A	Raman spectroscopy	Spectrum consistent with cocaine HCl reference material	Information provides Category A selectivity and the result is consistent with cocaine HCl

C	Color test	Positive color change consistent with cocaine HCl reference material	Information provides Category C selectivity and the result indicates cocaine or related compounds

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other**.** The scheme of Raman spectroscopy (Category A) and color test (Category C) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose. In addition, the Raman spectroscopy provided salt form information, which was not part of the question.

Conclusion: The sample contains cocaine.

Question #6A: Can I use a field-portable Raman instrument as a Category A test in Question 6?
Answer: The field-portable Raman instrument would have to be assessed and validated for this purpose. If the validation demonstrates the resolution and spectral range provides sufficient structural information to achieve the requisite selectivity requirement of an analogous Category A laboratory instrument determined to be fit for purpose and produces reviewable spectral data (see Part III B.5.1), then it may be used in the manner described in Scheme 6.

Question #7: Does the sample contain methcathinone?

Scheme Selected #7A: Time-of-flight mass spectrometry with Direct Analysis in Real Time ionization (DART-TOFMS) (Category A) + GC-FID (Category B)

Category	Technique	Result	Assessment
A	MS (DART-TOFMS)	$[M+H]^+$ ion (no fragmentation) consistent with methcathinone	Molecular ion and molecular formula information are consistent with methcathinone, but does not achieve Category A due to lack of structural information
B	GC-FID	t_R of analyte peak is consistent with methcathinone reference material	Information provides Category B selectivity and the result is consistent with methcathinone; another technique is required
Additional Technique: GC-MS (Category B + A)			
B	GC	t_R of analyte peak was not compared with a reference material	Comparison to reference material not performed; no information which could be assessed was obtained
A	MS (EI)	Spectrum of analyte is consistent with methcathinone reference material	Information provides Category A selectivity and the result is consistent with methcathinone

Discussion: The selected scheme of MS (DART-TOFMS) (Category A) and GC-FID (Category B) was insufficient to identify methcathinone. The GC-FID provided retention time information, but the DART-TOFMS did not provide fragmentation (structural information), therefore another technique (Category A, B or C) was necessary to obtain selectivity for methcathinone. In this example, a Category A (GC-MS (EI)) technique was chosen. The retention time from the hyphenated GC-MS test was not used.

The enhanced scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains methcathinone.

Question #7A: Would a DART-MS/MS have the same limitations as the DART-TOFMS in Question 7?
Answer: No. DART-MS/MS has the potential for increased selectivity by allowing for a selected precursor ion to be isolated and then individually fragmented, providing structural information for the analyte. The DART-MS/MS would have to be assessed and validated for this purpose to ensure that the fragmentation provides sufficient structural information to achieve the selectivity requirement of a Category A technique. DART-MS/MS differs from DART-TOFMS with in-source fragmentation. In DART-TOFMS with in-source fragmentation, the precursor ion cannot be selected in advance and the resulting fragmentation spectrum is a mixture of fragments of simultaneously generated precursor and fragment ions.

Scheme Selected #7B: GC-FTIR (Category B + A)

Category	Technique	Result	Assessment
B	GC	t_R of analyte peak is consistent with methcathinone reference material	Information provides Category B selectivity and the result is consistent with methcathinone
A	FTIR	Spectrum consistent with methcathinone reference material	Information provides Category A selectivity and the result is consistent with methcathinone

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other**.** The scheme of GC (Category B) and FTIR (Category A) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains methcathinone.

Question #8: Does the pharmaceutical preparation contain a controlled substance?

Scheme Selected #8A: Pharmaceutical identifier (Category C) + GC-FID (Category B) + TLC (Category B)

Category	Technique	Result	Assessment
C	Pharmaceutical identifier	Appearance consistent with a pharmaceutical-grade amphetamine sulfate tablet	Information provides Category C selectivity and indicates amphetamine sulfate tablet
B	GC-FID	t_R of analyte peak is consistent with amphetamine reference material	Information provides Category B selectivity and the result is consistent with amphetamine
B	TLC	R_f of analyte is consistent with amphetamine reference material	Information provides Category B selectivity and the result is consistent with amphetamine

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other. The scheme of pharmaceutical identifiers (Category C), GC-FID (Category B), and TLC (Category B) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains a controlled substance identified as amphetamine.

Scheme Selected #8B: Pharmaceutical identifier (Category C) + GC-MS (Category B + A)

Category	Technique	Result	Assessment
C	Pharmaceutical identifier	OC80 markings similar to a known pharmaceutical product containing oxycodone	Information provides Category C selectivity and indicates oxycodone tablet
B	GC	t_R of analyte peak is not consistent with oxycodone reference material, but is consistent with fentanyl reference material	Information provides Category B selectivity and the result is consistent with fentanyl
A	MS (EI)	Spectrum of analyte is too weak to provide sufficient information	Did not achieve Category A selectivity due to lack of structural information/low abundance
Additional Technique: GC-MS(EI) (Category A)			
-	GC	Data not required to affect fentanyl identification	

A	MS (EI) Concentrated sample	Spectrum of analyte is consistent with fentanyl reference material	Information provides Category A selectivity and the result is consistent with fentanyl

Discussion: The selected scheme of pharmaceutical identifier (Category C), GC-MS (Category B + A) was insufficient to identify oxycodone or fentanyl. The pharmaceutical identifier was inconsistent with the instrumental data and the initial mass spectrum did not provide sufficient sensitivity to obtain structural information, but the GC provided retention time information. The MS test was repeated after resampling and concentrating the sample.

The enhanced scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains fentanyl.

Scheme Selected #8C: Pharmaceutical identifier (Category C) + LC–UV/Vis Diode Array Detector (DAD) (Category B + B)

Category	Technique	Result	Assessment
C	Pharmaceutical identifier	Appearance consistent with a pharmaceutical-grade oxycodone tablet	Information provides Category C selectivity and indicates oxycodone tablet
B	LC	t_R of analyte peak is consistent with oxycodone reference material	Information provides Category B selectivity and the result is consistent with oxycodone
B	UV/Vis (DAD)	Spectrum consistent with oxycodone reference material	Information provides Category B selectivity and the result is consistent with oxycodone

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other. The scheme of pharmaceutical identifier (Category C), LC (Category B), and ultraviolet/visible spectroscopy (full spectrum) (Category B) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains oxycodone.

Scheme Selected #8D:
Initial Scheme: Pharmaceutical identifier (Category C) + GC-MS (Category B + A)
Final Scheme: GC-MS (Opioid Method; Category B + A) + GC-MS (Screening Method; Category B + A)
Note the initial GC-MS analysis used a targeted opioid method and the second analysis used a broad screening method which was suitable for the detection of low-level analytes.

Category	Technique	Result	Assessment
C	Pharmaceutical identifier	OC80 markings similar to a known pharmaceutical product containing oxycodone	Information provides Category C selectivity and indicates oxycodone tablet
B	GC	t_R of analyte peak is not consistent with oxycodone reference material, but is consistent with stearic acid	The result is not consistent with an oxycodone tablet but information provides Category B selectivity for stearic acid
A	MS (EI)	Spectrum obtained from analyte is consistent with stearic acid. No other substances detected on a targeted opioid method	Information provides Category A selectivity for stearic acid; however no controlled substances were indicated through analysis. Further testing required due to inconsistent test results
Additional Technique: GC-MS (EI) (Category B + A) using a concentrated extract and different acquisition parameters			
B	GC	No substances other than stearic acid detected when analyzed on a broad screening acquisition method	No information inconsistent with the original GC analysis was obtained
A	MS (EI)	No substances other than stearic acid detected when analyzed on a broad screening acquisition method	No information inconsistent with the original MS analysis was obtained

Discussion: The selected scheme of pharmaceutical identifier (Category C), GC-MS (Category B + A) was insufficient to support an identification of oxycodone or a finding of no controlled substances detected. The pharmaceutical identifier was inconsistent with the instrumental data acquired under routine conditions for suspected opioid containing tablets, which did not indicate any controlled substances. No controlled substances were detected through subsequent additional GC-MS analysis of a new concentrated sample analyzed using a broad screening acquisition method designed for the detection of low abundance analytes.

The modified scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: No controlled substances identified in the sample.

Question #9: Does the sample contain cannabis?
Scheme Selected #9: Macroscopic examination (Category B) + Microscopic examination (Category B) + Color test (Category C).
Note that the laboratory does not have access to a trained botanist, so the identification will be conducted by a drug chemist.

Category	Technique	Result	Assessment
B	Macroscopic examination	Characteristic morphological features of cannabis observed	Information provides Category B selectivity and the result is consistent with cannabis
B	Microscopic examination	Characteristic microscopic features of cannabis observed	Information provides Category B selectivity and the result is consistent with cannabis
C	Color test	Positive color change consistent with cannabinoids (e.g., THC, CBD,CBN)	Information provides Category C selectivity and the result indicates THC or other cannabinoids

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other. The scheme of macroscopic examination (Category B), microscopic examination (Category B), and color test (Category C) provides a scientifically supported conclusion to the question asked and is, therefore, fit for purpose.

Conclusion: The sample contains cannabis.

Question #9A: Would all of the tests in the selected scheme for Question 9 be necessary if the analysis was performed by a trained botanist?
Answer: No. If the analysis was performed by a trained botanist or an analyst appropriately trained in botanical identification (where jurisdiction allows), analysis of the morphological characteristics of cannabis following an established botanical analytical scheme is sufficient to provide a scientifically supported conclusion to the question asked. Therefore, the color test in Scheme 9 would not be necessary.

Question #9B: Is a suspected cannabis sample considered Marijuana in a jurisdiction where Marijuana is defined as the plant cannabis with a concentration of Δ9-THC greater than 0.3% by weight?
Note: since the jurisdictional definition of Marijuana cannabis includes a minimum Δ9-THC concentration, the analytical scheme must include a test that either measures the concentration of Δ9-THC or employs a decision point to determine if the Δ9-THC concentration exceeds the stated threshold.
Selected Scheme #9B: Macroscopic examination (Category B) + Microscopic examination (Category B) + Color test (Category C) + GC-MS (Category B + A)

Category	Technique	Result	Assessment

B	Macroscopic examination	Characteristic morphological features of cannabis observed	Information provides Category B selectivity and the result is consistent with cannabis
B	Microscopic examination	Characteristic microscopic features of cannabis observed	Information provides Category B selectivity and the result is consistent with cannabis
C	Color test	Positive color change consistent with cannabinoids with [THC] > [CBD]	Information provides Category C selectivity and the result indicates THC predominant Cannabis
B	GC (qualitative and semi-quantitative)	t_R of analyte peaks are consistent with Δ^9-THC and CBD; height of Δ^9-THC peak compared to internal standard indicates total Δ^9-THC greater than the assigned decision point value	Information provides Category B selectivity and the result supports Δ^9-THC and CBD, and the [Δ^9-THC] is greater than 0.3%
A	MS (EI)	Spectra consistent with Δ^9-THC and CBD,	Information provides Category A selectivity and the result are consistent with Δ^9-THC and CBD

Discussion: To meet jurisdictional requirements, an appropriately selective color test (Category C) as well as a GC-MS (Category B + A) method that includes comparison of peak heights with an internal standard. The concentration of the internal standard is a validated decision point value sufficiently greater than the statutory threshold after the application of measurement uncertainty. The method is validated to give a yes or no answer for the question whether the Δ9-THC concentration was greater than the statutory threshold of 0.3%.

The enhanced scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains Marijuana Cannabis.

Question #10: Can 4-methylmethcathinone (4-MMC) be identified in a sample without reference materials?
Note that known isomers of MMC include 2-MMC, 3-MMC and 4-MMC.
Scheme Selected #10: GC-MS (Category B + A)

Category	Technique	Result	Assessment
B	GC	t_R of analyte peak was not compared with a reference material	Comparison to reference material not performed; no information which could be assessed was obtained

A	MS (EI)	Spectrum consistent with MMC reference spectra in the SWGDRUG library. From the structural information provided, it is not possible to identify the positional isomer	Information provides Category A selectivity and the result is consistent with 2-, 3-, or 4-MMC; but does not provide sufficient selectivity to answer the question; another technique is needed
Additional Technique: NMR (Category A)			
A	NMR	Spectrum provides structural determination of 4-MMC from SWGDRUG monograph, and distinguishable from 2- and 3-MMC from SWGDRUG monographs	Information provides Category A selectivity and the result is consistent with 4-MMC

Discussion: The selected scheme of GC (Category B) and MS (Category A) was insufficient to identify 4-MMC. As no reference materials are available for contemporaneous comparison, the retention time from GC cannot be used toward the identification. Although the mass spectrum is consistent with the MMC reference spectrum in the SWGDRUG library, there is insufficient information to determine the specific isomer. Therefore, another technique (NMR - Category A) was chosen to identify the isomer.

The enhanced scheme provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains 4-methylmethcathinone. (For reporting guidance, see Parts IV A 6.1.6 and IV C 2.2.2)

Question #11: Does a liquid sample obtained contain any psychoactive substances?

Scheme Selected #11: Color tests (category C) + GC-MS (Category B + A)

Category	Technique	Result	Assessment
C	Color Tests	No color changes consistent with known controlled substances	Information does not provide Category C selectivity
B	GC	Methanol extract does not produce analyte peaks.	Information does not provide Category B selectivity
A	MS (EI)	No spectral information obtained.	Information does not provide Category A selectivity; another technique is needed
Additional Technique: GC-FID (Category B)			

B	GC	Concentrated basified chloroform extract does not produce analyte peaks on a broad screening program.	No inconsistent information was obtained.

Discussion: Initial testing using routine methodology did not indicate the presence of a controlled substance. An additional concentrated extract from a new sample in a different extraction solvent (acquired on a more sensitive instrument) was deemed to be appropriate for the sample type (to rule out the presence of a substance in salt form whose detection could only occur when present in free base form).The results obtained from this additional analysis supported those from the original analysis.

The enhanced scheme provides a scientifically supported conclusion to the question asked (noting the extractions used were determined to be appropriate for the sample) and, therefore, is fit for purpose.

Conclusion: No controlled substances identified in the liquid.

Question #12: Does the sample contain psilocin?
Scheme Selected #12: GC-MS (Category B + A) + Thin Layer Chromatography (Category B)

Category	Technique	Result	Assessment
B	GC	t_R of analyte peak is consistent with psilocin reference material	Information provides Category B selectivity and the result is consistent with psilocin. However, the source of the psilocin is unknown (from the material or as thermal degradation of psilocybin).
A	MS	Spectrum consistent with psilocin reference material	Information provides Category A selectivity and the result is consistent with psilocin. However, the source of the psilocin (from the material or as thermal degradation of psilocybin) is unknown, another technique is needed.
B	Thin Layer Chromatography	R_f of analyte spot is consistent with psilocin reference material and is inconsistent with psilocybin reference material	Information provides Category B selectivity and the result is consistent with psilocin

Discussion: The selected scheme of GC-MS (Category B + Category A) and Thin Layer Chromatography (Category B) was sufficient to identify psilocin. The MS test provided the required Category A structural information, but it could not determine the

source of the psilocin (whether from the material or as a thermal degradation of psilocybin). Therefore, another technique (Thin Layer Chromatography) was required.

The scheme of GC (Category B), MS (Category A), and Thin Layer Chromatography (Category B) provided a scientifically supported conclusion to the question asked and, therefore, was fit for purpose.

Conclusion: The sample contains psilocin.

Question #13: Does the sample contain psilocybin?
Scheme Selected #13: LC-MS/MS (Category B + A)

Category	Technique	Result	Assessment
B	LC	t_R of analyte peak is consistent with psilocybin reference material	Information provides Category B selectivity and the result is consistent with psilocybin
A	MS/MS	Fragmentation spectrum consistent with psilocybin reference material	Information provides Category A selectivity and the result is consistent with psilocybin

Discussion: Each technique achieves the level of selectivity required of its category and the positive test results corroborate each other**.** The scheme of LC (Category B) and MS/MS (Category A) provides a scientifically supported conclusion to the question asked and, therefore, is fit for purpose.

Conclusion: The sample contains psilocybin.

Clandestine Drug Laboratory Evidence

Additional considerations for analysis of chemicals found in Clandestine Drug Laboratory evidence is put forth in Part III C *Methods of Analysis / Clandestine Drug Laboratory Evidence*. For convenience of the reader, the Analytical Groups and techniques described in Part III C are included here as a reference.

Analytical Groups and Techniques[5]

[5] The list of analytical techniques is not exhaustive and is in no particular order.

Analytical Group	Techniques
Analytical Group 1 Elemental Analysis Techniques (IIIC.4.4.1)	• Atomic Absorption Spectroscopy • Atomic Emission Spectroscopy and Flame Tests (an attached spectrometer significantly increases the selectivity relative to flame tests) • Energy Dispersive X-Ray Detectors for Scanning Electron Microscopes (SEM-EDX) • Mass Spectrometry (utilizing Inductively Coupled Plasma sources or for elements with unique isotopic abundance patterns) • X-Ray Fluorescence (XRF)
Analytical Group 2 Structural Elucidation Techniques (IIIC.4.4.2)	• Infrared Spectroscopy (IR and FTIR) • Mass Spectrometry • Nuclear Magnetic Resonance (NMR) • Raman Spectroscopy • UV-Vis & Fluorescence Spectroscopy
Analytical Group 3 Separation Techniques (IIIC.4.4.3)	• Capillary Electrophoresis • Gas Chromatography • Ion Chromatography • Liquid Chromatography • Thin Layer Chromatography
Analytical Group 4 Chemical Properties (IIIC.4.4.4)	• Flammability • Microcrystalline tests • pH (of liquids or vapors) • Radioactive decay • Reactivity with water, air, or other materials • Solubility and miscibility tests • Spot and precipitation tests
Analytical Group 5 Physical Properties (IIIC.4.4.5)	• Color • Crystal forms measured with polarized light microscopy or x-ray diffraction techniques • Density (relative density and density of mixtures have reduced selectivity) • Phase transitions including melting points, boiling points, sublimation temperature and vapor pressure • Physical state or states • Refractive index • Viscosity and surface tension

Where possible, the identification of organic compounds shall follow the

recommendations for the analysis of seized drugs (see Part III B – Analytical Scheme for Identification of Drugs or Chemicals).

The selectivity of analytical techniques for the identification of inorganic materials depends on the particular analyte. In each case the analytical scheme shall:

- Have sufficient selectivity to identify the material to the exclusion of others (e.g., identification of both the cation and anion in salts)
- utilize two or more techniques, preferably from different analytical groups

Question #14A: Is this a sample of red phosphorus?
Scheme Selected #14A: SEM-EDX (Group 1) + Chemical Properties (Group 4)

Analytical Group	Technique	Result	Assessment
Group 1	SEM-EDX	The energy of the x-rays detected are consistent with those reported for phosphorus; no other responses detected	Elemental composition of the material is phosphorus but does not differentiate between phosphorus oxidation states
Group 4	Allotrope Conversion[6]	Heating a sample produced a puff of white fumes consistent with a change in allotropic form	Information provides Group 4 selectivity and the result is consistent with red phosphorus
Group 4	pH	Fumes produced from the allotrope conversion gave an acidic result to moistened pH paper, consistent with the production of phosphoric acid from white phosphorus.	Information provides Group 4 selectivity and the result is consistent with red phosphorus

Discussion: Each technique achieves the level of selectivity required of its analytical group and the positive test results corroborate each other. The scheme of SEM-EDX (Group 1) and Chemical Properties (Group 4) provides a scientifically supported conclusion to the question asked and is, therefore, fit for purpose.

Conclusion: The sample contains red phosphorus.

[6] McKibben, T, et. al., Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence. Journal of the Clandestine Laboratory Investigating Chemists Association. Vol. 5 Number 4. October 1995. P 19-33.

Note: *If the relevant legislation does not require the identification of the phosphorus allotrope present (e.g. red phosphorus, white phosphorus (also referred to as yellow phosphorus) or black phosphorus) then the substance can be identified as phosphorus and the Group 4 testing is not required.*

Question #14B: Is this sample of phosphorus viable for use in the production of hydroiodic acid or has the sample of phosphorus been used previously for the manufacture of methamphetamine?

Scheme Selected #14B: GC-MS (Groups 3 + 2)

<table>
<tr><th>Analytical Group</th><th>Technique</th><th>Result</th><th>Assessment</th></tr>
<tr><td>Group 3</td><td>GC</td><td>No substances were detected.</td><td>Information does not provide Category B or Group 3 selectivity.</td></tr>
<tr><td>Group 2</td><td>MS (EI)</td><td>No substances were detected. No spectral information obtained</td><td>Information provided does not provide Category A or Group 2 selectivity; additional testing needed.</td></tr>
<tr><td colspan="4">Additional technique: Heat sample and extract into isooctane for GC-MS analysis to confirm allotropic form.</td></tr>
<tr><td>Group 4</td><td>Allotrope Conversion</td><td>Allotropic conversion to white phosphorus indicated through GC-MS analysis</td><td rowspan="3">The information provided after the GC-MS analysis of the extracted reaction product provides selectivity from Group 2, 3, and 4 techniques and supports the identification of red phosphorus in the initial sample.</td></tr>
<tr><td>Group 3</td><td>GC</td><td>A single peak is present in the GC data</td></tr>
<tr><td>Group 2</td><td>MS (EI)</td><td>Spectrum of analyte is consistent with external reference data for white phosphorus</td></tr>
<tr><td colspan="4">Additional technique: React sample with iodine in the presence of methanol and heat to determine viability for reaction.</td></tr>
<tr><td>Group 4</td><td>React with iodine in methanol with heat.</td><td>Production of methyl iodide indicated through GC-MS analysis.</td><td rowspan="3">The information provided after the GC-MS analysis of the reaction product provides selectivity from Group 2, 3, and 4 techniques and supports the production of methyl iodide.</td></tr>
<tr><td>Group 3</td><td>GC (Headspace acquisition, no solvent delay)</td><td>t_R of analyte peak is consistent with methyl iodide reference data</td></tr>
<tr><td>Group 2</td><td>MS (EI)</td><td>Spectrum of analyte is consistent with external reference data for methyl iodide.</td></tr>
</table>

Discussion: The lack of white phosphorus in the first GC-MS indicates that white phosphorus was not present in the initial sample. The GC-MS testing does not provide an indication of use of the material in the manufacture of methamphetamine. The confirmation of white phosphorus after the allotropic conversion supports the presence of red phosphorus in the initial sample but does not provide information about its viability or use in the manufacture of methamphetamine. The addition of iodine to the sample (in the presence of a small amount of water) produced hydriodic acid, which reacts with methanol to produce methyl iodide. This was detected by Headspace GC-MS(EI), showing the red phosphorus in the initial sample was viable for further reaction.

The additional analysis provides the required information to form a scientifically supported conclusion to the question asked and is, therefore, fit for purpose.

Conclusion: The sample contains red phosphorus which is viable for use in the production of methamphetamine. There is no indication of previous use.

Question #15: What is contained in an unknown liquid?
Scheme Selected #15: Chemical Properties (Group 4)

<table>
<tr><th>Analytical Group</th><th>Technique</th><th>Result</th><th>Assessment</th></tr>
<tr><td>Group 4</td><td>pH</td><td>pH of 0 obtained</td><td>Information obtained provides Group 4 selectivity and indicates the solution is acidic.</td></tr>
<tr><td>Group 4</td><td>Spot Test – Reaction with Silver nitrate solution</td><td>Black precipitate formed</td><td>Information obtained provides Group 4 selectivity and indicates the solution contains a reducing acid.</td></tr>
<tr><td colspan="4">Additional technique: React sample with ammonium hydroxide in the presence of a reducing acid (Group 4) and analyze product by ATR-FTIR (Group 2).</td></tr>
<tr><td>Group 4</td><td>Reaction with ammonium hydroxide and evaporation of water.</td><td>White solid produced</td><td rowspan="2">Information obtained provides Group 4 and Group 2 selectivity which supports the presence of hypophosphite anions in the solution.</td></tr>
<tr><td>Group 2</td><td>ATR-FTIR</td><td>Spectrum obtained is consistent with reference data for ammonium hypophosphite</td></tr>
</table>

Discussion: The formation of a black precipitate indicates the reduction of Ag^{2+} to Ag which occurs in the presence of a reducing acid such as phosphorous acid or hypophosphorous acid. The additional testing demonstrates the presence of hypophosphite ions in the solution. The identification of ions allows the confirmation of the identity of the liquid.

The scheme chosen provides the required information to form a scientifically supported conclusion to the question asked and is, therefore, fit for purpose.

Conclusion: The sample contains hypophosphorous acid.

Question #16: Does the dark solid contain iodine?
Scheme Selected #16: Physical properties (Group 5) + Chemical Properties (Group 4) + GC-MS (Groups 3 + 2)

Analytical Group	Technique	Result	Assessment
Group 5	Physical Properties	The substance produces persistent brown staining on a piece of tissue paper	Information provides Group 5 selectivity and indicates the presence of iodine
Group 4	Reaction with hypophosphorous acid in methanol.	Production of methyl iodide indicated in GC-MS analysis.	The information provided after the GC-MS analysis of the reaction product provides selectivity from Group 2, 3, and 4 techniques and supports the production of methyl iodide.
Group 3	GC (Headspace acquisition, no solvent delay)	t_R of analyte peak is consistent with methyl iodide reference data	
Group 2	MS (EI)	Spectrum of analyte is consistent with reference data for methyl iodide.	

Discussion: The persistent brown staining produced by the solid is consistent with that produced by elemental iodine. The addition of hypophosphorous acid to the sample produced hydriodic acid, which then reacts with methanol to produce methyl iodide.

This scheme chosen provides the required information to form a scientifically supported conclusion to the question asked and is, therefore, fit for purpose.

Conclusion: The sample contains iodine.