

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 1 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

## 10.1 Trace Non-Routine Analysis

### 10.1.1 Introduction

The listed items under "10.0 Trace Evidence General Analysis" are a compilation of miscellaneous specialized analyses which do not appear in the other sub-discipline protocols in this manual.

The guidelines below will serve as a resource for the other non-routine types of analyses which may be requested of the trace chemist.

Items submitted for general chemical examinations are the miscellaneous samples not included in another examination (e.g. greases, glues, cosmetics, unknown substances). They are often referred to as any chemistry-related exams requested of the laboratory that are not a request for the identification of a controlled substance.

While the Trace unit sees some evidence types on a fairly routine basis, others may require method development at the time the case is being worked. No procedures manual could encompass methods for every general chemical evidence type analyzed. This section will provide analysis schemes for some of those that the Trace unit has analyzed with some regularity and will give guidance for those types not yet encountered as they are not normally an evidence type.

Note - Items that are suspected to contain a drug should be analyzed by the Controlled Substances Unit first, then sent to the Trace Evidence Unit only if no drugs are found, or if some additional non-drug adulterant is also suspected.

A drug is defined as:

A chemical that causes a physiological effect. This includes so-called recreational drugs, pharmaceuticals, botanicals, and fungi that are used because of known, alleged, or assumed effects. A drug does not have to be "controlled". This does not include insecticides, rodenticides, cyanide, ethylene glycol, inorganics, or antibiotics even though these may technically have a physiological effect.

Note – Items that are beverages requested to be checked for ethyl alcohol or requested to have ethyl alcohol quantitated should be analyzed by the Toxicology Unit.

#### 10.1.1.1 Possible Reasons for Submission:

- Identification of contents
- Determination if specific compound(s) is/are present
- Determination if a questioned substance is comparable to a known
- Routine case takes a turn

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 2 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

### 10.1.1.2 Possible Role of Analyses:

- Provide links between:
- Victim & Suspect
- Victim & Scene
- Suspect & Scene
- Provide proof of a criminal act.
- Provide investigative leads.
- Support or refute a story.
- Provide information for reconstructions.

### 10.1.1.3 Some Common Submissions

- Unknown powders
- Unknown liquids
- Unknown mixtures
- Items with unknown residues
- Solids (including food) thought to contain adulterants
- Liquids (including beverages) thought to contain adulterants

### 10.1.1.4 Some Common Analytes

- Inorganic salts
- Building materials
- Acids
- Bases
- Greases
- Oils
- Cleaning products
- Bleach
- Volatile organics
- Solvents
- Pesticides
- Random materials/chemicals

## 10.1.2 Minimum Standards & Controls for Miscellaneous Non-Routine Evidence

These are dependent upon the type of evidence being submitted and the examinations being performed. Generally, blanks, reference standards and controls are run along with the evidence items and all of these results are recorded in the case file notes.

If instrumentation is used, follow instrumentation minimum standards and controls.

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 3 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

### 10.1.3 Definitions

**Blank:** an analysis performed on a laboratory-prepared sample which includes all components of the unknown sample except for the material of interest. All reagents, preparatory steps and analysis conditions will be identical to that used on the unknown sample.

**Control:** a test performed on a known sample under identical conditions as that used on the unknown sample.

**Minimum Standard of Analysis:** the minimum required analytical tests to be performed in order to report a conclusive finding.

**Preliminary Test:** an analytical procedure that yields information about a sample but by itself cannot be used as a basis for a conclusive finding.

**Confirmatory Test:** an analytical procedure that will specifically identify an unknown sample.

**Standards File:** a collection of spectra or chromatograms of known materials to be used as a reference for comparison to an unknown sample.

**Reagent:** any substance used in a reaction for the purpose of detecting, measuring, or analyzing other substances.

### 10.1.4 Instrumentation

See 7.0 Instrument Standards and Controls

### 10.1.5 General Requirements

- Blanks must be run at the same conditions as the samples. A blank will be run if the preceding instrument run was a positive result.
- All instrument hard copies generated from case exhibits must contain the case number, exhibit number, analyst's initials and date.
- Pertinent instrument parameter used for an analysis must be documented in the Procedures manual or in the case record in Forensic Advantage (FA).
- All calibration data must be documented and contain the analyst's initials and date.
- Identification or classification made by comparison to other than in-house standards must be noted in the case record.
- Identification or classification made via a library reference search must be confirmed by visual comparison to the library standard and included in the case record.

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 4 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

### 10.1.6 Documentation and Report Wording

Casefile Notes:

- State all tests performed and their results.
- All tests not described in the procedures manual must be fully documented. Reference documents used shall be documented in the work notes.
- An accurate description of the evidence will be recorded.
- If the sample is consumed in analysis, a notation will be made.
- All documentation is included to support the conclusion made. Report Wording:\* Report wording is to be as consistent as possible with previously reported exams of a given type.
- When reporting conclusions based upon the examination of "non-routine" evidence types the Supervisor, Technical Leader and/or technical reviewer will help ensure that the report wording is accurate, clear and concise.

### 10.1.7 Safety Considerations

It is the responsibility of the user of any protocol to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Proper caution should be exercised and the use of personal protective equipment should be considered to avoid exposure to dangerous chemicals. Consult the appropriate MSDS for each chemical prior to use.

### 10.1.8 Analytical Approach

Good scientific principles and a logical analysis scheme, along with appropriate consultations with the Supervisor or Technical Leader, are applied to those evidence types that have not been encountered before based upon the type and amount of evidence submitted and the case information received.

Generally speaking, a visual macroscopic/ microscopic examination will be performed (stereo, compound, comparison and polarizing microscopes) for color, texture, viscosity, morphology, optical properties and homogeneity.

Record the weight or volume of the evidence as received, if possible or applicable. Otherwise describe the amount received (e.g. trace amount of off-white powder, a few drops of green liquid).

Analytical approaches will vary depending upon the amount and type of sample received. With a limited amount of sample, nondestructive testing is performed first.

Solubility/miscibility tests with organic/inorganic and polar/nonpolar solvents (e.g. water, carbon disulfide, pentane, acetone, chloroform) may be done.

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 5 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

The pH of unknown liquids and their corresponding controls, as well as water soluble/miscible samples may be taken using pH paper and followed up using a pH meter if available or applicable.

Microchemical tests may be performed as indicators of a class of compounds present (e.g. for sulfates, chlorides, nitrates, sugars). Necessary instrumentation is used to identify or to compare samples.

Acid/base extractions, as well as extractions based on solubility can be performed to partially or totally separate the components of a mixture prior to instrumental analysis.

- Suggested acid/base extraction protocol:

- approx 6 ml sample in tube + conc. H<sub>2</sub>SO<sub>4</sub> to acidic pH, mix, + 7-8 ml CH<sub>2</sub>Cl<sub>2</sub>, mix, centrifuge, draw off organic layer (bottom), filter through Na<sub>2</sub>SO<sub>4</sub> on cotton in pipette, dry, + 0.5 ml CH<sub>2</sub>Cl<sub>2</sub> (acid extract)
- Make aqueous basic w/conc. NaOH to basic pH, mix, + 8-9 ml CH<sub>2</sub>Cl<sub>2</sub>, mix, centrifuge, draw off organic layer (bottom), filter through Na<sub>2</sub>SO<sub>4</sub> on cotton in pipette, dry, + 0.5 ml CH<sub>2</sub>Cl<sub>2</sub> (base extract)
- Air dry 2<sup>nd</sup> approx. 6 ml sample, add 0.5 ml MeOH, mix, draw off clear (MeOH extract)

It may not be possible to fully identify an unknown. However, general classification is usually possible. When comparison samples are submitted or obtained, it may be possible to say that the unknown shares the same physical and/or chemical properties as the comparison sample.

### **Example Approaches to the Analysis of General Unknowns (see below):**

Start with general approach and compile information.

Narrow down possibilities with ultimate goal of identification.

- General Observations
- Screening Techniques
- Microscopy
- Molecular Identification

Analytes can be broken down into solids, liquids, and gases, each of which may contain single components, homogenous mixtures (e.g. liquid solutions), or heterogeneous mixtures (e.g. mixed crystalline compounds)

### **General Observations:**

- Packaging (commercial container, condition of container, listed ingredients, markings, etc.)
- State of sample (solid, liquid, gas, mixture)

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 6 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

- General amount of sample present
- Obvious odor

### 10.1.8.1 Solids - types of analysis techniques

- Visual/General Examination
  - Color
  - Consistency (e.g. metallic, powder, resin, polymeric, etc.)
- Stereomicroscopic Examination
  - Crystalline vs. Amorphous
  - Organic vs. Inorganic
  - Homogenous vs. Heterogeneous
- Manual separation of particles
- Light Microscopy/Polarized Light Microscopy
  - Color with transmitted light
  - Homogenous vs. Heterogeneous
  - Isotropic vs. Anisotropic
  - Presence of pigments/fillers
  - General refractive index
  - Particle identification
- Ignition test (low explosives, improvised explosive mixtures)
- Chemical Tests
  - Solubility
  - Spot Tests/Color Tests
  - Crystal Tests
  - Extractions
- pH (with addition of deionized water)
- Instrumentation:
  - XRF, SEM-EDS (Elemental)
  - GC, GC/MS, TOF-DART MS (Organics)
  - FTIR (including microscope, ATR, gas cell, liquid cell)
  - Ion Chromatography

#### General Approach for Solids:

- 1) Visual/General Examination
- 2) Stereomicroscopic Examination
- 3) PLM
- 4) Elemental Analysis
- 5) Compound Identification
  - a. FTIR
  - b. Mass Spectrometry (GC/MS, Py-GC/MS)

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 7 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

6) Additional tests

- a. Chemical Tests
- b. Ignition Tests

**10.1.8.2 Liquids**

- Visual/General Examination
  - Color
  - Single vs. Multi-phase (organic/aqueous mix)
  - General viscosity
  - Presence of any precipitate or sediment
  - pH
  - Conductivity (Ionic Solution)
  - Ignition Test (Ignitable Liquid)
  - Solid-Liquid Solution (Solids in a Liquid)
  - Liquid-Liquid Solution
- Extractions
  - Liquid-Liquid
  - Acid/Base
  - Solid Phase Extraction
  - Precipitation
  - Evaporation/Distillation
  - Heated and Passive Headspace (volatile organics)
- Chemical tests
  - Precipitate Reactions
  - Spot Tests
  - Color Tests
  - Crystal tests
- Instrumental Analysis
  - Elemental Analysis
  - XRF (in absence of vacuum)
  - SEM-EDS (precipitates only)
  - GC, GC/MS (organic solutions, extracts)
  - FTIR
  - Liquid samples (gas cell, liquid cell, ATR)
  - Precipitates
  - Ion Chromatography

**General Approach for liquids:**

- 1) Visual/General Examination
- 2) pH

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	Document #: 7438	Page 8 of 9
	Revision #: 2	Issued Date: 07/06/2020
	Document Manager: Cheryl Lozen	Approved By: Ryan Larrison

- 3) Conductivity
- 4) Ignition Test
- 5) Extraction/Solvent Dilution/Evaporation/Precipitation
- 6) Sample Screening/Identification of Components
  - a) FTIR
    - b) Mass Spectrometry (GC/MS)
    - c) Elemental Analysis
- 7) Chemical Spot Tests

#### **10.1.8.3 Gases**

- Consider Safety Concerns - Contact FSD Health and Safety Officer
- Analysis
  - GC, GC/MS
  - FTIR via Gas Cell

#### **10.1.8.4 Elemental Analysis**

Organic vs. Inorganic  
 Elemental composition  
 Preliminary information for screening purposes  
 SEM-EDS vs. EDXRF

#### **10.1.9 Resources/References**

NIST Chemistry WebBook <http://webbook.nist.gov/chemistry/>

Household Products Database <http://householdproducts.nlm.nih.gov/>

Chamot, Emil and Mason, Clyde, Handbook of Chemical Microscopy. 2nd Edition. Chicago: McCrone Research Institute, 1989

Fiegl and Anger, Spot Tests in Inorganic Analysis. 6th Edition. Cambridge: Elsevier Publishing Co., 1972

	<b>TRACE-PM 10.1 Trace Non-Routine Analysis</b>	
	<i>Document #: 7438</i>	<i>Page 9 of 9</i>
	<i>Revision #: 2</i>	<i>Issued Date: 07/06/2020</i>
	<i>Document Manager: Cheryl Lozen</i>	<i>Approved By: Ryan Larrison</i>

Jungreis, Ervin, Spot Test Analysis-Clinical, Environmental, Forensic, and Geochemical Applications. New York: John Wiley & Sons, 1985

McCrone and Delly, The Particle Atlas., 2nd Edition. Ann Arbor: Ann Arbor Science, 1973

The Merck Index, Twelfth Edition (or later), 1996