

# #16

## General Unknowns Analytical Method

### 1.0.0 Background and Scope

There are times when samples do not fit into a certain category. These procedures are designed to analyze these samples, examples of which are pills, liquid pharmaceuticals, and samples that do not give the expected results with screening tests. Whenever possible, two different tests, and two different sampling events will be employed in confirming the presence of controlled substances. One of the tests must provide structural information, i.e. either MS or FTIR.

### 2.0.0 Equipment and Reagents

The following pieces of equipment can be used in any combination to identify the analytes of interest.

- 2.1.0 A GC/MS and appropriate analytical software. Reference GC/MS Analytical Method.
- 2.2.0 FTIR and appropriate analytical software. Reference Gendrug AM section 10.
- 2.3.0 ACS grade solvents.
- 2.4.0 0-14 pH paper.
- 2.5.0 n-Tridecane internal standard. In the ratio of 1.3ml tridecane into 1 L chloroform.

### 3.0.0 Sample Preparation and Analysis

This section details the minimum requirements for analysis, any additional testing if necessary may be performed at the analyst discretion. As per the General Drug AM section 9.2.0 a positive color test can always be used in lieu of a second GC/MS run or in conjunction with the FTIR in order to satisfy the second sampling event rule. This applies to both solid and liquid samples. While performing testing using two GC/MS sampling events, if the first GC/MS run is negative the second sampling event does not need to be analyzed. Any extract with internal standard must be returned with the evidence.

#### 3.1.0 Solids

- 3.1.1 Extractions Using appropriate sampling, extract with methanol, chloroform, or a mixture of methanol and chloroform. Liquids that are approximately as viscous as honey can be treated as solids.
- 3.1.2 Acidic, basic, neutral, and dry extractions, in any combination, can be employed in order to separate diluents or other interferences.

#### 3.2.0 Liquids.

- 3.2.1 If possible, determine if the sample is aqueous, this may be done through (immiscibility or flame) testing (preferred) or from information submitted with the sample. If the sample is organic go to 3.2.6. If it is not possible to determine if the sample is aqueous then treat the sample as if it is.
- 3.2.2 If possible split the sample and use a basic extraction using approximately 0.5N sodium carbonate or 0.5N sodium bicarbonate and an immiscible solvent, chloroform preferred. If the sample cannot be split, add internal standard to the solvent before analysis on the GC/MS.
- 3.2.3 Alternatively, an aliquot may be evaporated with air/nitrogen and either

- reconstituted with methanol and/or a methanol-chloroform mixture and analyzed on the GC/MS or the residue may be analyzed using the FTIR .
- 3.2.4 Refer to general drug AM 2.8.0 for reporting conclusions.
  - 3.2.5 Organic solvents may be analyzed directly with the GC/MS. Split the sample if possible and add internal standard if necessary. If after the first GC/MS analysis the sample needs to be diluted, dilute with methanol and reanalyze. The sample may be diluted with methanol before analysis if the analyst has reason to expect the sample to be concentrated.
  - 3.2.6 Meth lab samples may be cleaned up using back extractions and solvent exchange. Steroid samples may be diluted with an appropriate solvent or another clean-up method may be used.
  - 3.2.7 If a sample is suspected of containing a controlled substance that is covered by a separate method, i.e. GHB, then that method should be used.
- 3.3.0 Analysis.
- 3.3.1 Run samples using a general unknown data acquisition method.
  - 3.3.2 If a peak appears, and is not recognized, perform a library search.
  - 3.3.3 If a controlled substance is recognized from a library search or other means, then a standard is run if identity is to be confirmed. Library search reports do not need to be retained in the case file.
- 3.4.0 Conclusions.
- 3.4.1 Confirmation. The retention time must be within 0.040 min of a valid scan of the standard and the MS spectra must match. If both conditions are satisfied then confirmation can be reported.
  - 3.4.2 Non-confirmation. If a standard is not available but the library search produces a match then report "Results of testing indicates the presence of a controlled substance, not confirmed". The reason why the substance is not confirmed must be on the report.
  - 3.4.3 If the RT or MS do not match, or there is no peak at all, then report, "No controlled substances detected".
  - 3.4.4 As with all cases it is up to the analyst to decide whether or not to report non-controlled substances.

#### **4.0.0 FTIR Sample Preparation and Analysis Methods**

- 4.1.0 Direct.  
Samples may be analyzed directly with the ATR. Samples may also be mixed with KBr, pressed into a pellet/window and then analyzed.
- 4.2.0 Extractions
  - 4.2.1 The organic layer from either a basic or acidic extraction may be mixed with ground KBr, evaporated and analyzed.
  - 4.2.2 Samples undergoing a basic extraction may require bubbling with HCl gas and filtering before HCl salt can be isolated and analyzed.
- 4.3.0 Analysis
  - 4.3.1 Analyze samples per the General Drug AM section 10.
  - 4.3.2 Perform a library or literature search of the resulting spectra.
- 4.4.0 Conclusions
  - 4.4.1 Confirmation.  
If the spectra of the standard in the ISP Forensics produced library and sample match in all significant respects the compound may be reported.

- 4.4.2 Non-confirmation  
If a spectra from an ISP Forensics produced library is not available but the library or literature search produces a match the presence of the compound may be reported with a “not confirmed” statement.
- 4.4.3 If a spectral match to a controlled substance is not made then the sample must be analyzed on the GC/MS.
- 4.4.4 The analyst may decide whether or not to report non-controlled substances.

## 5.0.0 History

<b>Revision #</b>	<b>Issue or review date</b>	<b>History</b>	<b>Author or Reviewer</b>
0	9/20/02	Original Issue	D.C. Sincerbeaux
1	3/13/03	Rev sec 3.2	D.C. Sincerbeaux
2	1/12/07	Rev sec 4.2.1, 3.14	D.C. Sincerbeaux
3	7/2/12	Changed 2.2.0, 3.1.4.3, 3.3.2, 4.1.0 4.3.1, 4.4.3	D.C. Sincerbeaux
4	2/22/13	Changed 3.1.4, 3.1.4.2, 3.3.2	D.C. Sincerbeaux
5	12/1/14	Changed all of section 3, added 2.5.0	D.C. Sincerbeaux
6	12/16/14	Changed 3.0.0, 3.1.0, 3.2.0, 3.2.1, 3.2.1.5, 3.2.2, 3.2.3.1, 3.2.4, 3.2.5, 3.2.6 (added by number change), 3.3.1 renumbered most of 3.0.0.	D.C. Sincerbeaux
7	6/10/15	Changed 3.0.0, 3.2.0, 3.2.4, 3.2.5 Added 3.2.6 and 3.2.7 Changed numbering in 3	D.C. Sincerbeaux
8	12/30/15	Dropped 3.2.3 renumbered section.	D.C. Sincerbeaux