

Idaho State Police

Forensic Services

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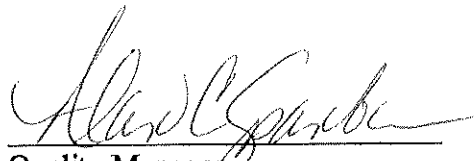


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Quality Manager


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#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 FTIR Analytical Method.
- 2.3.0 ACS grade solvents.
- 2.4.0 pH paper

3.0.0 GC/MS Sample Preparation and Analysis

3.1.0 Extraction.

- 3.1.1 Solids. Using appropriate sampling, extract with methanol, or a mixture of methanol and chloroform.
- 3.1.2 Acidic and/or basic extractions can be employed in order to separate diluents or other interferences. These extractions can be performed with commercial products (Toxi-Lab tubes) or laboratory generated solutions.
- 3.1.3 Organic solvents may be diluted or injected directly.
- 3.1.4 Aqueous samples. With the exception of Clanlab samples, aqueous samples not giving a positive screening test and having a negative result for a controlled substance after a basic extract, will be analyzed using an acidic extraction, assuming adequate sample size.
 - 3.1.4.1 Determine initial pH. Split sample and adjust pH accordingly.
 - 3.1.4.2 Extract with immiscible solvent.
 - 3.1.4.3 If the sample is of limited volume dilute with methanol, approximately 10:1, methanol to sample.

3.2.0 Analysis.

- 3.2.1 Run samples using a general unknown data acquisition method.
- 3.2.2 If a peak appears, perform a library search.
- 3.2.3 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.

- 3.3.0 Conclusions.
 - 3.3.1 Confirmation. The retention time must be within 0.04 min of a valid scan of the standard and the MS spectra must match. If both conditions are satisfied then confirmation can be reported.
 - 3.3.2 Non-confirmation. If a standard is not available but the library search produces a match then report the presence of the compound with a “not confirmed” statement.
 - 3.3.3 If the RT or MS do not match, or there is no peak at all, then report, “No controlled substances detected”.
 - 3.3.4 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.

Powders may be ground with KBr and run directly or analyzed using the microscope attachment. Organic liquid samples may be placed on a window of KBr or a commercial equivalent.
- 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 FTIR AM.
 - 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 no spectral match is found report the sample as “No controlled substances detected”.
 - 4.4.4 The analyst may decide whether or not to report non-controlled substances.

5.0.0 History

<u>Revision #</u>	<u>Issue or review date</u>	<u>History</u>	<u>Author or Reviewer</u>
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 Added #	D.C. Sincerbeaux