#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 GendrugAM section 10.
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 base/neutral extract, will be analyzed using an acidic extraction, assuming adequate sample size. Samples submitted as controls will be treated the same as the samples they were submitted with.
  - 3.1.4.1 Determine initial pH. Split sample and adjust pH accordingly.
  - 3.1.4.2 Extract with immiscible solvent. If the sample is neutral then it may be evaporated with air/nitrogen and reconstituted with methanol/chloroform.

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.

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3.3.2 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.

3.3.3 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.

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 section10.

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

Revision #	Issue or review date	History	Author or Reviewer
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	2/25/13	Changed 3.1.4, 3.1.4.2, 3.1.4.3, 3.3.2	D.C.Sincerbeaux