History for the Phenethylamine SOP

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Phenethylamine Family
Standard Operating Procedures

1.0.0 Background

The phenethylamine family of drugs include, but are not limited to, amphetamine, methamphetamine, phentermine, ephedrine, pseudoephedrine, 3,4-MDA, and MDMA. Almost all are controlled substances, or can be used to make controlled substances, and can be identified by several different techniques currently used by the ISP-Forensic Service laboratories. General information can be found in numerous articles (Microgram etc.) and books such as "Drug Identification Bible", 4th edition, 1999.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of phenethylamines in samples. 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, NMR, or FTIR.

3.0.0 Equipment and Reagents

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

3.1.0 A GC/MS and appropriate analytical software. Reference GC/MS SOP.
3.2.0 FTIR and appropriate analytical software. Reference FTIR SOP.
3.3.0 Polarizing microscope and reagents. Reference General Drug SOP.

4.0.0 Color Spot Tests

Marquis, Liebermanns, and sodium nitroprusside (2nd Amines) are some of the most common spot tests used to help identify the phenethylamines. Recipes for these, and other reagents can be found in "Clarke's Isolation and Identification of Drugs" 2nd Edition, 1986.

5.0.0 GC/MS Sample Preparation and Analysis

Either of two extraction methods can be used depending on the analyst's discretion.

5.1.1 Basic Extraction. Place approximately 0.05g of sample into a test tube. Dissolve with distilled water. Make basic with Na2CO3 or another strong base. Extract with petroleum ether, hexane, or other non-water soluble solvent. Analyze on GC/MS. The standards are prepared using this method. **NOTE** Amphetamine and methamphetamine basic extracts are volatile. If the extract in the sample vial is allowed to completely evaporate then the analyte may be lost. It is important to recap the sample vial with a new septa if the extract needs to be saved for reanalysis or returned to evidence in a trace case where all of the original sample was used.

5.1.2 Direct extraction. A small amount of the sample, approximately 0.01 g, is dissolved in methanol or other appropriate solvent, and analyzed on the GC/MS.

5.2.0 GC/MS analysis. The retention time of the sample should be within 0.04 min of a valid MS scan from the daily standard. **NOTE** Underivatized ephedrine and pseudoephedrine cannot be separated using normal GC/MS columns. To identify either separately, the sample must be analyzed using a FTIR.

6.0.0 FTIR Sample Preparation Methods

When purification is necessary the following methods should be used in making KBr pellets. Alternative sample introduction techniques can be used when appropriate.

6.1.0 Amphetamine and Methamphetamine.

6.1.1 Dissolve sample in water or dilute acid. Make basic with Na2CO3, or another strong base, and extract with petroleum ether or hexane. Wash extract with water then dry through Na2SO4. Bubble HCl gas through solvent and collect the resulting crystals. Wash with additional petroleum ether and let dry. Make KBr pellet. Compare against a known HCl salt standard.

6.1.2 dl-Methamphetamine by PIT derivative.
Dissolve sample in water and make basic with Na2CO3. Extract with petroleum ether and dry extract through NaSO4. Add 2 drops of phenylisothiocyanate(PIT) and let stand for 10 minutes. Decant solvent and wash crystals with additional solvent. Dry and make a KBr pellet. Compare with a dl-Methamphetamine standard that was prepared using this procedure.

6.1.3 Direct. Mix and grind sample with KBr. Form into a pellet.

6.2.0 Ephedrine and Pseudoephedrine.

6.2.1 Using the same procedure as 6.1.1 will yield the HCl salt.

6.2.2 To obtain the base form, dissolve the sample in water, make basic and extract with petroleum ether or hexane. Evaporate the solvent and make a KBr pellet. Compare against a known base standard.

6.2.3 If the sample is a pill(s), crush and add methanol and shake well. Wait approximately one hour and centrifuge. Place supernatant into a clean test tube and allow too evaporate. Take crystals from side of test tube and make a KBr pellet.