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1. AMPHETAMINE AND RELATED PHENETHYLAMINES

- A. Phenethylamines is a generic name for chemicals consisting of a phenyl group with a two-carbon chain amine. The carbon chain, the phenyl group and the amine may all have various substitutions. The drug action of these chemicals will vary based on the substitutions present. The most common substituted phenethylamines analyzed by the Controlled Substances Section include amphetamine, methamphetamine, ephedrine/pseudoephedrine, MDA, and MDMA. However, any sample with this general chemical structure may be analyzed according to this procedure and available literature information.
- B. Structure, Empirical Formula, Molecular Weight

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HN CH ₃
MMCH3

 $\begin{array}{c} \text{Methamphetamine} \\ C_{10}H_{15}N \\ \text{MW 149.2} \end{array}$

Phentermine $C_{10}H_{15}N$ MW 149.2

3,4-Methylenedioxyamphetamine (MDA)
$$\begin{array}{c} C_{10}H_{13}NO_2\\ MW\ 179.2 \end{array}$$

Methcathinone C₁₀H₁₃NO MW 163.2

Mescaline C₁₁H₁₇NO₃ MW 211.3

3,4-Methylenedioxymethamphetamine (MDMA) $C_{11}H_{15}NO_2 \\ MW~193.2$

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3,4-Methylenedioxy-N-ethylamphetamine (MDEA) $C_{12}H_{17}NO_2$ MW 207.2

p-Methoxymethamphetamine (PMMA) C₁₁H₁₇NO MW 179.2

4-Methyl-2,5-dimethoxyamphetamine (STP)

$$C_{12}H_{19}NO_2$$
 MW 209.3

MW 165.2

4-Bromo-2,5-dimethoxyphenethylamine (Nexus) $C_{10}H_{14}BrNO_2$ MW 260.13

3-methoxy-4,5-Methylenedioxyamphetamine (MMDA) $C_{11}H_{15}NO_{3}$ MW 209.2

Name	Formula	Molecular Weight	Trade Names	Drug Action
Amphetamine	C ₉ H ₁₃ N	135.2	Adderall®	CNS Stimulant
Cathine	C ₉ H ₁₃ NO	151.21		CNS Stimulant

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Diethylpropion	C ₁₃ H ₁₉ NO	205.3	Tenuate®, Tepanil®	CNS Stimulant
N,N-Dimethylamphetamine	C ₁₁ H ₁₇ N	163.26		CNS Stimulant
Ephedrine/ Pseudoephedrine	C ₁₀ H ₁₅ NO	165.2	Benadryl®, Sinutab®, Sudafed®, etc.	CNS Stimulant
N-Ethylamphetamine	C ₁₁ H ₁₇ N	163.26		CNS Stimulant
Methamphetamine	C ₁₀ H ₁₅ N	149.2	Desoxyn®	CNS Stimulant
Methcathinone	C ₁₀ H ₁₃ NO	163.22		CNS Stimulant
Phentermine	C ₁₀ H ₁₅ N	149.24	Adipex-P®, Fastin®	CNS Stimulant
Phenylpropanolamine	C ₉ H ₁₃ NO	151.2	Removed from market	CNS Stimulant
3,4-Methylenedioxyamphetamine (MDA)	C ₁₀ H ₁₃ NO ₂	179.2		Hallucinogen and stimulant
3,4-Methylenedioxymethamphetamine (MDMA)	C ₁₁ H ₁₅ NO ₂	193.2		Hallucinogen
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	C ₁₂ H ₁₇ NO ₂	207.27		Hallucinogen
3-methoxy-4,5-Methylenedioxyamphetamine (MMDA)	C ₁₁ H ₁₅ NO ₃	209.2		Hallucinogen
Mescaline	C ₁₁ H ₁₇ NO ₃	211.3		Hallucinogen
p-Methoxyamphetamine (PMA)	C ₁₀ H ₁₅ NO	165.2		Hallucinogen
p-Methoxymethamphetamine (PMMA)	C ₁₁ H ₁₇ NO	179.2		Hallucinogen
4-Methyl-2,5-dimethoxyamphetamine (STP)	C ₁₂ H ₁₉ NO ₂	209.3		Hallucinogen
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	C ₁₀ H ₁₄ BrNO ₂	260.13		Hallucinogen

C. Synonyms

Name	Synonyms
Amphetamine	White crosses, α -methylbenzeneethaneamine, phenylisopropylamine, dextroamphetamine
Cathine	Norpseudoephedrine, katine

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Diethylpropion	∞-benzoyltriethylamine, 2-(diethylamino)-1-phenyl-1-propanone, amfepramone
N,N-Dimethylamphetamine	N-methylmethamphetamine, N,N,∞-Trimethylbenzeneethaneamine
Ephedrine/ Pseudoephedrine	Ma huang, Sudafed
N-Ethylamphetamine	Ethamphetamine
Methamphetamine	Speed, meth, glass, ice, crank, N,α -dimethylbenzeneethaneamine, desoxyephedrine, d-methamphetamine, methylamphetamine
Methcathinone	∞-methylaminopropiophenone, ephedrone, cat, goob
Phentermine	∞,∞ -Dimethybenzeneethaneamine, ∞ -benzylisopropylamine, phenyl- <i>tert</i> -butylamine, robin's eggs, black and whites
Phenylpropanolamine	PPA, dl-norephedrine, 1-phenyl-2-amino-1-propanol
3,4-Methylenedioxyamphetamine (MDA)	MDA, ∞-methyl-3,4-(methylenedioxy)-phenethylamine, methylenedioxyamphetamine, "Mellow Drug of America", Love
3,4-Methylenedioxymethamphetamine (MDMA)	MDMA, XTC, Adam, ecstasy, E, 3,4-methylenedioxymethylamphetamine
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	MDE, MDEA, Eve, N-ethyl-3,4-methylenedioxyamphetamine
3-methoxy-4,5-Methylenedioxyamphetamine (MMDA)	MMDA, 5-(∞-methyl)ethanamine-6-methoxy-1,3-benzodioxole
Mescaline	3,4,5-trimethoxybenzeneethanamine, 3,4,5-trimethyoxyphenethylamine
p-Methoxyamphetamine (PMA)	4-methoxyamphetamine
p-Methoxymethamphetamine (PMMA)	4-methoxymethamphetamine, 4-methoxymethylamphetamine
4-Methyl-2,5-dimethoxyamphetamine (STP)	2,5-dimethoxy-4-methylamphetamine, 4-methyl-2,5-dimethoxyphenylisopropylamine, STP, DOM
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	Nexus, 2-CB, 4-bromo-2,5-dimethoxybenzeneethaneamine

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D. Common pharmaceutical/street forms

Name	Common pharmaceutical/street forms
Amphetamine	5, 10, 20, and 30 mg tablets. Used in the treatment of attention deficit hyperactivity disorder, (ADHA ADHD), in children and in the treatment of narcolepsy.
	Clandestine manufacture produces crystalline powder of various colors, which may be mixed with methamphetamine, possibly due to the starting chemicals in the reaction mixture. It may also be encountered in liquids, slurries of various colors, or coffee filters. Other common street forms include syringes containing liquid and amphetamine impregnated cottons.
Cathine/Cathinone	Present in <i>Catha edulis</i> , (Khat plants), used for social and medicinal purposes in East Africa and Southern Arabia.
	Fresh Khat plants contain both cathinone and cathine. Upon sitting at room temperature, cathinone decomposes to cathine. Fresh Khat plants should be stabilized upon receipt into the lab by freezing.
Diethylpropion	25-75 mg tablets. Used as an appetite suppressant/anorexic.
N,N-Dimethylamphetamine	No medicinal use in the U.S.A.
Ephedrine/ Pseudoephedrine	Contained in tablets and syrups, which can be over the counter products or prescribed by a physician. Both are ingredients in antihistamines, decongestants, diet aides, and bronchial asthma medication.
	It may be extracted from these preparations and used as the direct precursor in the clandestine manufacture of methamphetamine.
	In the clandestine manufacture of methamphetamine they may be encountered in tablets, powders, liquids, slurries of various colors, and coffee filters.
N-Ethylamphetamine	Historically used as an appetite suppressant/anorexic. No current medicinal use in the U.S.A.

Methamphetamine	5, 10, and 15 mg tablets. Used in the treatment of attention deficit
	hyperactivity disorder, (ADHA ADHD), in children and as an appetite

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	suppressant/anorexic in the treatment of severe obesity.
	Clandestine manufacture produces crystalline powder of various colors or large clear crystals. It may also be encountered in liquids, slurries of various colors, or coffee filters. Other common street forms include syringes containing liquid and methamphetamine impregnated cottons.
Methcathinone	Clandestinely manufactured. Illicitly sold primarily as the hydrochloride salt.
Phentermine	15-37.5 mg capsules and tablets. Used as an appetite suppressant/anorexic.
Phenylpropanolamine	Removed from market in 2001 by the FDA. Previously in prescription and over the counter diet and cold medications.
3,4-Methylenedioxyamphetamine (MDA)	Pressed tablets, especially with custom logo imprints, and powder. No medicinal use in the U.S.A.
3,4-Methylenedioxymethamphetamine (MDMA)	Pressed tablets, especially with custom logo imprints, or powder. No medicinal use in the U.S.A.
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	Pressed tablets, especially with custom logo imprints, and powder. No medicinal use in the U.S.A.
3-methoxy-4,5-Methylenedioxyamphetamine (MMDA)	Powder or pressed tablets. No medicinal use in the U.S.A.
Mescaline	Mescaline is the active constituent in peyote buttons. For information regarding peyote, see peyote monograph. No medicinal use in the U.S.A.
p-Methoxyamphetamine (PMA)	Pressed tablets, especially with custom logo imprints, and powder. No medicinal use in the U.S.A.
p-Methoxymethamphetamine (PMMA)	Pressed tablets, especially with custom logo imprints, and powder. No medicinal use in the U.S.A.
4-Methyl-2,5-dimethoxyamphetamine (STP)	Capsules. No medicinal use in the U.S.A.
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	Pressed tablets and powder. No medicinal use in the U.S.A.

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E. Solubility

Name	Solubility
Amphetamine	Base: Diethyl ether, methanol, chloroform, acids, slightly soluble in water
	Salt: Water, ethanol, methanol, insol. in diethyl ether, chloroform
Cathine	Diethyl ether, methanol, ethanol, chloroform, dilute acids
Diethylpropion	Salt: Water, part. Soluble in chloroform, methanol, insol. In diethyl ether
N,N-Dimethylamphetamine	Base: Diethyl ether, methanol, chloroform, water
Ephedrine/ Pseudoephedrine	Base: Diethyl ether, methanol, chloroform, slightly sol. in water Salt: Water, methanol, slightly sol. in chloroform
N-Ethylamphetamine	Diethyl ether, methanol, chloroform, water
Methamphetamine	Base: Diethyl ether, methanol, chloroform, acids, slightly sol. in water
	Salt: Water, methanol, chloroform, insol. in diethyl ether
Methcathinone	Water, methanol
Phentermine	Base: Diethyl ether, methanol, ethanol, chloroform Salt: Water, slightly sol. in chloroform, insol. in diethyl ether
Phenylpropanolamine	Salt: Water, ethanol, insol. in chloroform and ether
3,4-Methylenedioxy amphetamine (MDA)	Base: Diethyl ether, methanol, chloroform, dilute acetic acid
3,4- Methylenedioxymethamphetamine (MDMA)	Base: Diethyl ether, methanol, chloroform, dilute acetic acid
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	Base: Diethyl ether, methanol, chloroform, dilute acetic acid
3-methoxy-4,5-Methylenedioxyamphetamine (MMDA)	Base: Diethyl ether, methanol, chloroform, dilute acetic acid
Mescaline	Water, methanol, ethanol, chloroform, insol. in diethyl ether

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p-Methoxyamphetamine (PMA)	Base: Chloroform, dilute acids, insol. in water
	Salt: Water
p-Methoxymethamphetamine (PMMA)	Base: Chloroform, dilute acids, insol. in water
	Salt: Water
4-Methyl-2,5-dimethoxyamphetamine (STP)	Base: Chloroform
	Salt: Water
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	Salt: Water, methanol

F. Chemical indicator tests, crystal tests, extractions and TLC

Name	Marquis	NP	Lieb	Chen's	H ₂ SO ₄	Mecke's	Froehde's
Amphetamine	Org→ brn	NR	Red-org	NR			
Cathine	Yel→or g	NR	Yel	NR	Yel		
Diethylpropion	NR	NR	Yel	NR	NR		
N,N-Dimethylamphetamine	Org→ brn		Red-org				
Ephedrine/ Pseudoephedrine	NR	NR	Yel	Lavend er			
N-Ethylamphetamine	Org→ brn	Blue	Red-org				
Methamphetamine	Org→ brn	Blue	Red-org	NR			
Methcathinone	NR	NR	Lt Yel	NR			
Phentermine	Org→br n	Slow Blue	Red-org	NR	NR		
Phenylpropanolamine	NR	NR		Lavend er			

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3,4-Methylenedioxy amphetamine (MDA)	Purple	NR	Grn-blk	NR	Purple	Grn→blue -blk	Blue-blk
3,4- Methylenedioxymethamphetamine (MDMA)	Purple- blk	Blue	Grn-blk	NR	Purple	Blue-grn	Blue-blk
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	Purple- blk	Slow Blue	Grn	NR	Purple	Blue-grn	Grn→ blue-blk
3-methoxy-4,5- Methylenedioxyamphetamine (MMDA)	Brn	NR	Yel-grn		Yel-org	Yel-brn	Brn-blk
Mescaline	Org	NR	Blk			Brn	Blk
p-Methoxyamphetamine (PMA)	NR	NR	Purple- brn			Weak Olive	Weak grn
p-Methoxymethamphetamine (PMMA)	NR	Blue	Purple- brn			Weak Olive	Weak grn
4-Methyl-2,5-dimethoxyamphetamine (STP)	Yel	NR	Grn-brn			Yel-grn w/org ppt.	Yel-grn→ grn →brn
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	Yel→Grn	NR		NR	Yel		

Name	Common extractions	TLC Mobile Phase 14a	TLC Locator 14b
Amphetamine	2, 3, 4, 5, 6, 7, 8	i, ii	i, ii, iii
Cathine	9	i, ii	iv
Diethylpropion	1, 2, 3	i, ii	i, ii, iii
N,N-Dimethylamphetamine	3		
Ephedrine/ Pseudoephedrine	2, 3, 4, 5, 6, 7, 8, 10	i, ii	i, ii, iii, iv (pseudo)
N-Ethylamphetamine	3		
Methamphetamine	2, 3, 4, 5, 6, 7, 8	i, ii	i, ii
Methcathinone	1, 3	iii	V

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Phentermine	1, 2, 3	i, ii	i
Phenypropanolamine	2, 3, 4, 5, 6, 7, 8,	i, ii	i, ii, iii
3,4-Methylenedioxy amphetamine (MDA)	3	i, ii, iii	iv
3,4- Methylenedioxymethamphetamine (MDMA)	3	i, ii, iii	i
3,4-Methylenedioxy-N-Ethylamphetamine (MDEA)	3	i, ii, iii	i
3-methoxy-4,5-Methylenedioxyamphetamine (MMDA)	3		
Mescaline	1, 3	i, ii	i, ii, iii, v
p-Methoxyamphetamine (PMA)	3		
p-Methoxymethamphetamine (PMMA)	3		
4-Methyl-2,5-dimethoxyamphetamine (STP)	3	i, ii	i, ii, iii
4-Bromo-2,5-dimethoxyphenethylamine (Nexus)	1, 3		

(1) Dry extraction.

Note: If poor chromatography occurs, the samples may be basified and extracted as in (2) or (3).

- (a) Dissolve in C15 methanol
- (b) Filter to remove any insoluble material if necessary.

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- (2) Liquid/liquid extraction for pharmaceutical preparations
 - (a) Determine amount of sample required for extraction based on the concentration in the sample.
 - (b) Basify with 10% sodium hydroxide.
 - (c) Extract with diethyl ether.
 - (d) Evaporate diethyl ether and reconstitute with C15 methanol.
- (3) Liquid/liquid extraction for illicit powders/tablets
 - (a) Basify with 10% sodium hydroxide.
 - (b) Extract with diethyl ether.
 - (c) Evaporate diethyl ether and reconstitute with C15 methanol.
- (4) Liquid/liquid extraction from aqueous solutions
 - (a) Basify with 10% sodium hydroxide.
 - (b) Extract with diethyl ether.
 - (c) Evaporate diethyl ether and reconstitute with C15 methanol.
- (5) Liquid/liquid extraction from organic solutions
 - (a) Extract organic solution with 10% hydrochloric acid.
 - (b) Remove hydrochloric acid layer and basify with 10% sodium hydroxide.
 - (c) Verify pH is basic.
 - (d) Extract with diethyl ether.
 - (e) Evaporate diethyl ether and reconstitute in C15 methanol.
- (6) Liquid/liquid extraction from slurried pill extraction
 - (a) Remove a small amount of the slurried material.
 - (b) Basify with 10% sodium hydroxide.
 - (c) Extract with diethyl ether.
 - (d) Evaporate diethyl ether and reconstitute with C15 methanol.

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- (7) Liquid/liquid extraction from coffee filters
 - (a) Place a small piece of the coffee filter in a test tube.
 - (b) Add 10% sodium hydroxide to cover.
 - (c) Extract with diethyl ether.
 - (d) Evaporate diethyl ether and reconstitute with C15 methanol.
- (8) Optional liquid/liquid extraction for samples contaminated with blood
 - (a) Place approximately 3 drops of liquid containing blood into a spot plate.
 - (b) Add 1 drop of concentrated ammonium hydroxide or 10% sodium hydroxide.
 - (c) Add approximately 0.5 milliliters of chloroform.
 - (d) Mix thoroughly.
 - (e) Remove chloroform to a clean well.
 - (f) Evaporate 1-2 drops of chloroform for each chemical indicator test.
 - (g) Evaporate the remaining chloroform and reconstitute in C15 methanol for GC/MS analysis. See also CS-SOP-64.

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(9) Extraction of Cathine/Cathinone from Khat plants

Note: Fresh Khat plants contain cathinone and cathine. If stored at room temperature, cathinone decomposes to cathine. Fresh Khat plants can be stabilized by freezing. Cathine and cathinone known standards should be base/ether extracted for the GC/MS.

- (a) Place approximately 2 grams of fresh leaf material and tender stems or approximately 2 grams of dried leaf material in a beaker.
- (b) Add sufficient 0.1 N hydrochloric acid to cover plant material and sonicate for at least 15 minutes.
- (c) Decant the acid into a clean, new vessel.
- (d) Adjust the pH to above 9 with 2N sodium hydroxide.
- (e) Extract twice with chloroform.
- (f) Dry the chloroform layer with anhydrous sodium sulfate as needed.
- (g) Filter or decant the chloroform and evaporate to **near** dryness. **Do not evaporate completely to dryness.** Derivatization can be done at this point if desired.
- (h) Reconstitute in caff/MeOH if derivatization will be performed. If no derivatization will be performed reconstitute in C15/MeOH.

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- (10) Derivatization of ephedrine/pseudoephedrine:
 - (a) Extract as appropriate from above (2-8).
 - (b) Transfer solvent to a glass screw top test tube and evaporate.
 - (c) Dissolve residue in 200 μ L ethyl acetate.
 - (d) Add 200 μL pentafluoropropionic anhydride, (PFPA).
 - (e) Cap tightly and place in oven or heating block set at 80°C for 30 minutes.
 - (f) Cool to room temperature.
 - (g) Evaporate sample to dryness under air or nitrogen at 45 °C or lower.
 - (h) Reconstitute in methanol.
- (11) Cyclohexanone derivatization of khat extract or ephedrine/pseudoephedrine
 - (a) Use the GC/MS solution run on DRUGS1 from the khat extract or ephedrine/pseudoephedrine sample for the derivatization. This sample must be in caff/MeOH.
 - (b) Adjust the concentration of the solution to 1:1 (or more dilute) caffeine:cathine/PPA or caffeine:ephedrine/pseudoephedrine. Only use caff/MeOH.
 - (c) Add 40uL of the adjusted solution to a new GC/MS vial
 - (d) Add 40uL of 2% HCl in MeOH
 - (e) Dry the solution down under N2
 - (f) Add 400uL cyclohexanone
 - (g) Transfer solution to a glass insert
 - (h) Run on the GC/MS using the CYCLOHEX method
 - (i) Evaluate the chromatogram in the 7-8 minute time window
 - (j) Run several MeOH blanks after analysis to remove any cyclohexanone from the system
 - (k) Run the derivatized standards the same day as the khat extraction or ephedrine/pseudoephedrine sample

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- (12) Cyclohexanone derivatization standard preparation
 - (a) Use 20uL of a 1mg/mL standard solution in MeOH
 - (b) Dry the solution down under N2
 - (c) Add 20uL of 2mg/mL caffeine in cyclohexanone
 - (d) Add 80uL of cyclohexanone
 - (e) Transfer solution to a glass insert
 - (f) Run on the GC/MS using the CYCLOHEX method
 - (g) Compare standard mass spectrum to literature mass spectrum (El-Haj et al).
 - (h) PPA/cathine and ephedrine/pseudoephedrine have the same mass spectrum, but different retention times
 - (i) Cathinone mass spectrum should be compared to the data in the validation binder. Cathinone produces secondary peaks when derivatized
- (13) Procedure for Prepping Samples Containing MDMA and TFMPP for BSTFA with 1% TMCS Derivatization
 - (a) Determine the amount of sample required based on concentration of the sample.
 - (b) Basify with 10% sodium hydroxide.
 - (c) Extract with caffeinated chloroform or alternatively diethyl ether (evaporate diethyl ether and reconstitute in caffeinated chloroform).
 - (d) Add an equal amount of BSTFA with 1% TMCS.
 - (e) Place in GC vial, and cap.
 - (f) Heat for a minimum of 30 min at 200°C on the hot plate **or** 30 min at ~92°C in the heat block.
 - (g) Sample is ready to run on GC/MS DRUGS1B program.

NOTE: if GC/MS shows more underivitized compounds than derivitized, heat for an additional 30 min.

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(14) TLC:

- (a) Mobile Phase:
 - (i) System 1: Methanol:conc. ammonium hydroxide (100:1.5)
 - (ii) System 2: Chloroform:methanol (90:10)
 - (iii) System 3: Ethyl acetate:methanol:conc. ammonium hydroxide (85:10:5)
- (b) Locator:
 - (i) Acidified iodoplatinate reagent
 - (ii) Dragendorff reagent
 - (iii) Marquis reagent
 - (iv) Acidified potassium permanganate reagent
 - (v) Ninhydrin reagent
- G. GC/MS: Analyze using "Drugs1" program.

Notes:

<u>Ephedrine</u> and <u>pseudoephedrine</u> are positional isomers. However, they can be differentiated using GC retention times if they are derivatized, see extraction scheme 10 above. The samples must be compared to recent derivatized standards of ephedrine and pseudoephedrine.

Chromatography and/or separation between <u>cathine</u> and <u>cathinone</u> may be improved by derivatizing with TMS or acetic anhydride.

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H.	Report as:			
	"	", a dangerous drug.	See exceptions and examples be	elow.

Exceptions:

Ephedrine/pseudoephedrine, a precursor chemical I.

Phenylpropanolamine, a precursor chemical I.

p-Methoxymethamphetamine is currently not controlled under Arizona Law.

Examples:

Methamphetamine a dangerous drug in a usable condition.

Ephedrine/pseudoephedrine, a precursor chemical I.

3,4-methylenedioxymethamphetamine (MDMA) a dangerous drug in a usable condition.

Methylenedioxyamphetamine (MDA) a dangerous drug in a usable condition.

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