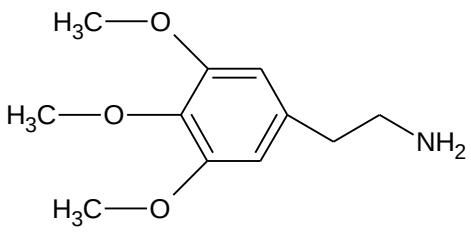
PEYOTE & MESCALINE

Latest Revision: June 27, 2005



1. SYNONYMS

CFR:

Mescaline
Peyote

CAS #:

Base: 54-04-6
Hydrochloride: 832-92-8
Sulfate: 1152-76-7

Other Names:

3,4,5-Trimethoxyphenethylamine
3,4,5-Trimethoxybenzeneethanamine
Lophophara willisamsii
Anhalonium lewinii
Mescal
Peyotl
Mezcline

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{11}H_{17}NO_3$	211.3	35-36
Hydrochloride	$C_{11}H_{17}NO_3 \cdot HCl$	247.8	181
Sulfate dihydrate	$(C_{11}H_{17}NO_3)_2 \cdot H2SO_4\ 2H_2O$	540.7	183-186

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	S	S	I	I	S	S
Hydrochloride	I	****	I	I	S	S
Sulfate dihydrate	I	I	I	I	SS	PS

A = acetone, C = chloroform, E = ether, H = hexane, M = methanol and W = water, VS = very soluble, FS = freely soluble, S = soluble, PS = sparingly soluble, SS = slightly soluble, VSS = very slightly soluble and I = insoluble

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Marquis	Orange
Liebermann's	Black
Froehde's	Brown fading to colorless
Mandelin's	Green to violet to grey
Mecke's	Greenish brown changing to brown
Vitali's	Dull red; Purple/dull red/brown

3.2. CRYSTAL TESTS

REAGENT	CRYSTALS FORMED
Wagenaar's	Long, curved branching needles
Picric Acid	Very long rods
Mercuric Chloride	Bunches of long needles
Gold Chloride	Rhombic prisms
Potassium bismuth iodide	Dense rosettes

3.3. THIN LAYER CHROMATOGRAPHY

Mobile phase – strong ammonia solution:methanol (1.5:100) should equilibrate for one hour. Mescaline Rf is 0.22.[2]

Visualization

Acidified iodoplatinate spray.

3.4. GAS CHROMATOGRAPHY

Method MES-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% Phenyl / 95% Methyl Siloxane 12 m x 200 µm ID x 0.33 µm film thickness

Carrier gas: Hydrogen at 1.0 mL/min

Temperatures: Injector: 270°C
Detector: 280°C
Oven program:
1) 175°C initial temperature for 1.0 min
2) Ramp to 280°C at 15°C/min
3) Hold final temperature for 4.0 min

Injection Parameters: Split Ratio = 60:1, 1 µL injected

Samples are to be dissolved in methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.30	ketamine	1.45
methamphetamine	0.32	lidocaine	1.51
pseudoephedrine	0.46	2CI	1.56
MDA	0.60	aminopyrine	1.62
MDMA	0.68	2C-T-7	1.85
benzocaine	0.74	procaine	1.92
acetaminophen	0.96	cocaine	2.44
phenacetin	0.99	O6-monoacetylmorphine	3.28

mescaline	**1.00 (2.00 min)**	heroin	3.58
caffeine	1.37	quinine	7.98

Method MES-GCS2

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% Phenyl/95% Methyl Siloxane 30 m x 250 µm ID x 0.25 µm film thickness

Carrier gas: Helium at 1.0 mL/min

Temperatures: Injector: 200°C
Detector: 280°C
Oven program:
1) 100°C initial temperature
2) Ramp to 280°C at 20°C/min
3) Hold final temperature for 1.0 min

Injection Parameters: Split Ratio = 80:1, 1 µL injected

Sample Preparation:
1) Mix crushed and ground peyote into an aqueous 10% sodium bicarbonate solution and extract with an excess of chloroform. Remove the chloroform layer and centrifuge or filter the aqueous layer (10% sodium bicarbonate solution with ground peyote) to recover the plant material. Add methanol to the recovered plant material. Filter the methanol extract prior to analysis.
2) Mix crushed and ground peyote into an aqueous 10% sodium bicarbonate solution for several minutes. Filter the solution and retain the plant material. Add methanol to the recovered plant material. Filter the methanol extract prior to analysis.

COMPOUND	RRT	COMPOUND	RRT
mescaline	**1.00 (7.12 min)**	anhalonidine	1.12
pellotine	1.11	lophophorine	1.14

3.5. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method MES-LCS1

Instrument: High performance liquid chromatograph equipped with diode array

Column: Waters Symmetryshield RP18 (4.5 x 150 mm, 3.5 µm

Detector: UV, 210 nm

Flow: 1.0 mL/min

Injection Volume: 5 µL

Buffer: 20mM Sodium Phosphate Monobasic pH = 2.3 with 0.2% Hexylamine (v/v)

Mobile Phase: 100% buffer for 4 min. Ramp for 2 min to 95% buffer : 5% acetonitrile, and hold for 6 min

Samples are to be dissolved in 0.1 N HCl, run buffer or methanol then filtered with a 0.45-micron filter.

COMPOUND	RRT	COMPOUND	RRT
ephedrine	0.44	MDA	0.87
pseudoephedrine	0.48	MDMA	0.91
amphetamine	0.59	**mescaline**	**1.00 (9.37 min)**
methamphetamine	0.71	MDEA	1.08

3.6. CAPILLARY ELECTROPHORESIS

Method MES-CES1

Internal Standard Stock Solution:
0.50 mg/mL procaine hydrochloride in 3.75mM sodium phosphate monobasic pH = 3.2.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of mescaline sulfate at 0.2 mg/mL using above internal standard stock solution.

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask and dilute with internal standard stock solution. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Mode:	Dynamically Coated Capillary Electrophoresis
Column:	60.2 cm x 50 µm fused silica capillary (50 cm to detector)
Run Buffer:	Microsolv DEA custom chiral run buffer
Detector:	UV, 195 nm
Voltage:	30 kV
Temperature:	20°C liquid cooled
Injection:	6.2 s hydrodynamic injection of sample at 0.5 psi 5.0 s hydrodynamic injection of water at 0.2 psi
Run Time:	13 min
Rinse Time:	0.1 N NaOH for 1 min at 50 psi Water for 1 min at 50 psi Microsolv CElixer A for 1 min at 50 psi Microsolv DEA custom chiral for 2 min at 50 psi

COMPOUND	RMT	COMPOUND	RMT
mescaline	**1.00 (8.25 min)**	d-methamphetamine	1.18
l-pseudoephedrine	1.07	procaine	1.29
d-ephedrine	1.11	l or d – MDA	1.36
l-amphetamine	1.13	l or d – MDA	1.37
l-ephedrine	1.13	l or d – MDMA	1.38

d-amphetamine	1.14	l or d – MDMA	1.40
l-methamphetamine	1.15		

4. SEPARATION TECHNIQUES

The procedure for extracting mescaline should begin with dried ground peyote buttons. The alkaloids are leached from the plant material by heating the plant material with ethanol or methanol and ammonium hydroxide. The extracts are filtered and evaporated just to dryness on a steam bath. Dissolve the residue in chloroform and 0.5N hydrochloric acid and extract the acid with additional chloroform, and wash the chloroform with acid. Discard the chloroform. Combine the acid fractions and add sodium carbonate to pH 8, then extract with chloroform.

Extract the phenolic alkaloids from the chloroform with 0.5N sodium hydroxide, filter, and evaporate the chloroform. Discard the sodium hydroxide. Dissolve the mescaline in alcohol, add sulfuric acid to neutral point. Filter mescaline sulfate and wash with cold alcohol.

Conversion of mescaline sulfate to mescaline hydrochloride is accomplished by first dissolving the sulfate in water. Using a strong base (NaOH or KOH), adjust the pH of the solution to around 10. Extract the resulting mescaline base into methylene chloride three times, each with equal volumes of solvent. Keep the mescaline base in the CH_2Cl_2 to prevent the formation of carbonate salts from exposure to atmospheric CO_2. Add 1N HCl to the sample and mix thoroughly; make sure the pH of the aqueous layer is ~2. The hydrochloride salt form remains in the aqueous layer. Remove the aqueous layer and evaporate the water.

Grind several grams of Dry Ice in a blender, add the dry sample (20-30 g), and grind to a fine powder. Weigh about 20 g powdered sample into a Soxhlet thimble. Cover with 95% alcohol, add 1 mL NH_4OH, and extract with alcohol for 3 hr. Let stand overnight (thimble covered with alcohol) and continue extraction for 3 more hours. Filter the extract through paper and evaporate just to dryness on a steam bath.

With the aid of CHC13 and 0.5N HC1, dissolve and transfer the residue to a separatory funnel. Shake and transfer the CHC13 to a second separatory funnel. Extract with 2 additional portions of HC1 and wash the aqueous solutions serially with fresh CHC13. Discard the CHC13 washings.

Carefully add solid Na2CO3 in small portions to the combined aqueous solutions until effervescence ceases, finally adjusting to pH 8 (check with indicator paper). Extract with four 50 mL portions of CHC13. Extract the combined CHC13 twice with ~ 0.5N NaOH to remove phenolic alkaloids, filter the CHC13, and concentrate to ~ 30 mL. Transfer to a 50 mL volumetric flask and adjust to volume. Withdraw a 2 mL aliquot for gas chromatography and evaporate the remainder just to dryness on a steam bath. Dissolve the residue in ~ 10 mL 95% alcohol and add H_2SO_4 (1 + 2) drop wise until neutral (check by touching glass rod to moist indicator paper). Crystalline mescaline sulfate will then separate. Cool, filter, and wash with cold alcohol.

Evaporate the filtrate to ~ 5 mL and add 1-2 drops 2N HCl. Crystalline anhalonine hydrochloride will separate. Filter and wash with cold alcohol.

To recover the phenolic alkaloids, acidify the NaOH extract obtained above, wash with $CHC1_3$, adjust to ~ pH 8 with solid Na_2CO_3, and extract with $CHC1_3$.

5. QUANTITATIVE PROCEDURE

5.1. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method MES-LCQ1

Internal Standard Solution:
95% 20mM sodium phosphate monobasic pH = 2.3 with 0.2% Hexylamine (v/v) : 5% Acetonitrile containing 0.3 mg/mL resorcinol.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of mescaline sulfate at approximately 0.5 mg/mL dissolved in the internal standard solution. Filter solution through a 0.45-micron syringe filter.

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask and dilute to volume with the internal standard solution. If necessary, dilute the sample so the final concentration approximates the standard concentration or falls within the linear range. Filter solution through a 0.45-micron filter.

Instrument:	High performance liquid chromatograph equipped with diode array
Column:	Waters Symmetryshield RP18 (4.5 x 150 mm, 3.5 µm)
Detector:	UV, 210 nm
Flow:	1.5 mL/min
Injection Volume:	2.0 µL
Buffer:	20 mM Sodium Phosphate Monobasic pH = 2.3 with 0.2% Hexylamine (v/v)
Mobile Phase:	Buffer: acetonitrile 95:5
Typical Retention Time:	Mescaline: min
Linear Range:	0.10 – 0.8 mg/mL
Repeatability:	RSD less than 1%
Correlation Coefficient:	0.9999
Accuracy:	Error less than 5%

COMPOUND	RRT
mescaline	**1.00 (2.74 min)**
resorcinol	1.97

Note: This method is for the quantitation of tablets, capsules, and powders containing mescaline. This method has not been validated for the quantitation of mescaline in plant material.

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)
Aqueous Acid	268

See spectra on the following pages for FT-IR, Mass Spectrometry, Nuclear Magnetic Resonance, and Vapor Phase IR.

7. REFERENCES

Bamford, F., Poisons: Their Isolation and Identification, 3rd Edition, Revised by C.P. Stewart, 1951.

Budavari, S., *The Merck Index, 12th Edition*, Merck and Co., Inc., 1996.

Clandestine Laboratory Guide for Agents and Chemists.

Clarke, E.G.C, *Isolation and Identification of Drugs, Vol. I*, The Pharmaceutical Press, 1969.

Clarke, E.G.C, *Isolation and Identification of Drugs, 2nd Edition*, The Pharmaceutical Press, 1986.

Levine, J., Microgram 1968, 1(4): 28.

Maloney, D.C., Microgram 2001, 34(8): 205

Mills III, T. and Roberson, J. C., *Instrumental Data for Drug Analysis*, 2nd Edition, Volume 2, Elsevier 1987, p.1359.

8. ADDITIONAL RESOURCES

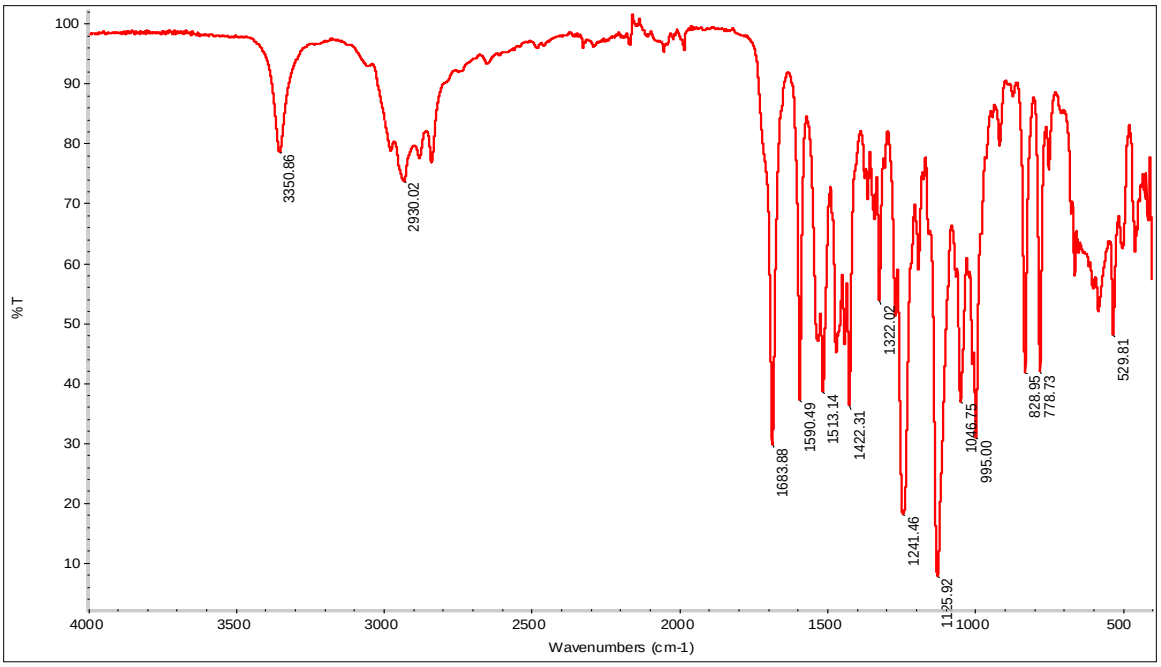
Forendex

Modified anonymous communication from www.erowid.org.

Wikipedia

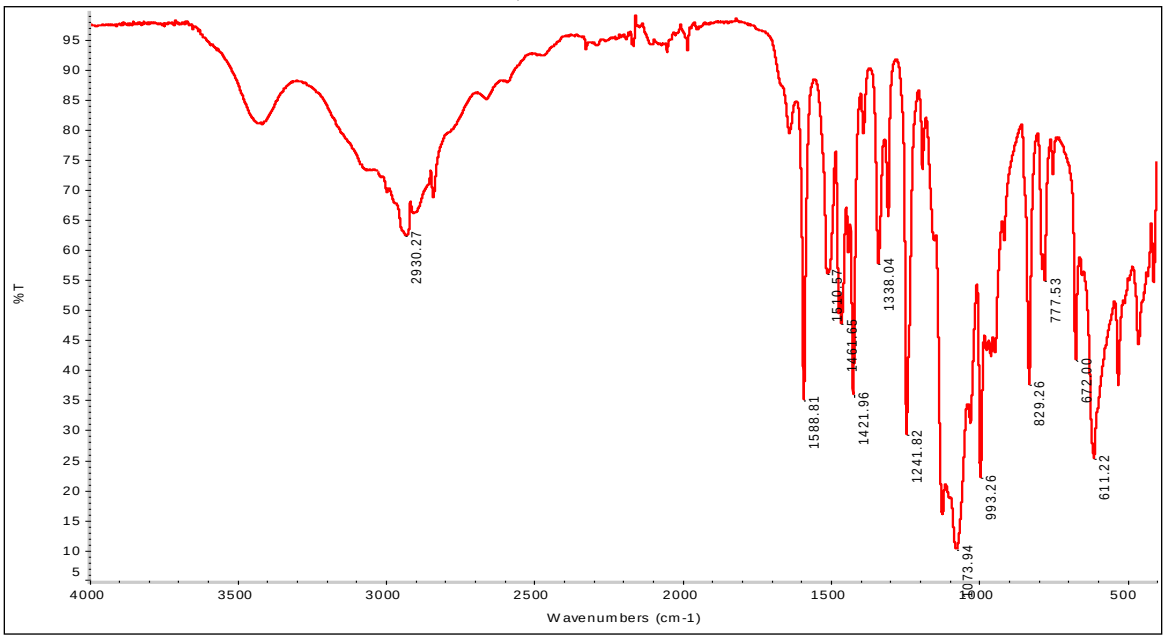
FTIR (Sample on single bounce ATR): Mescaline Base

32 scans, 4cm^{-1} resolution

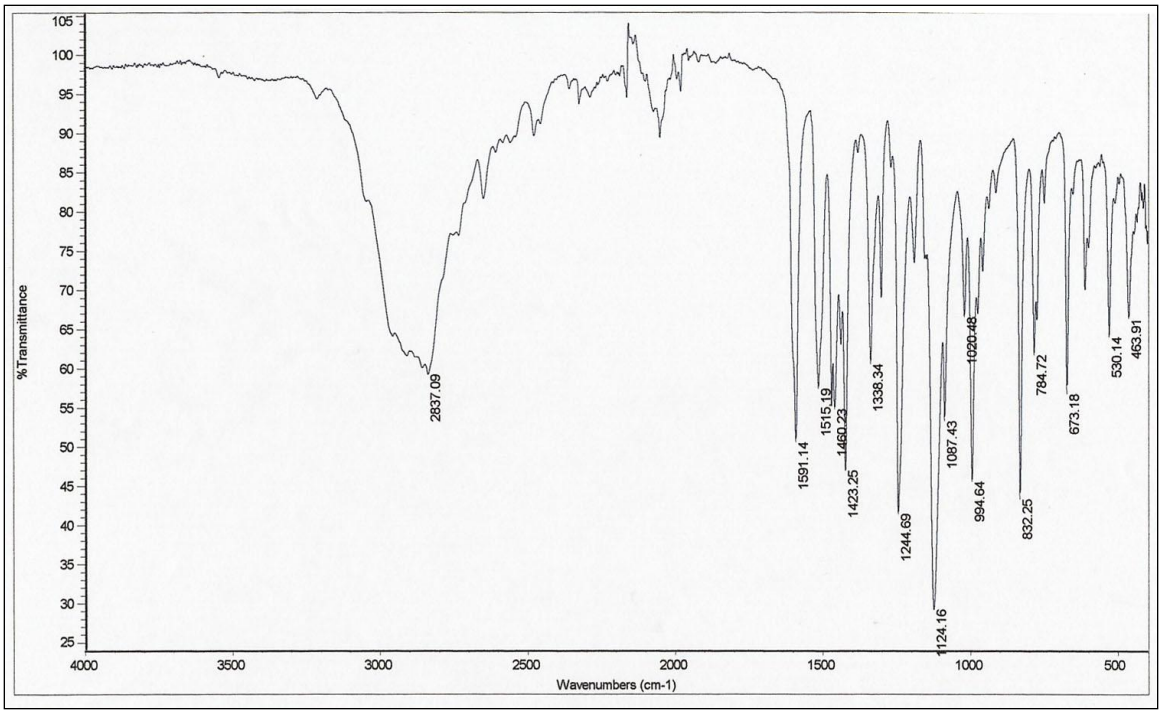


FTIR (Sample on single bounce ATR): Mescaline Sulfate

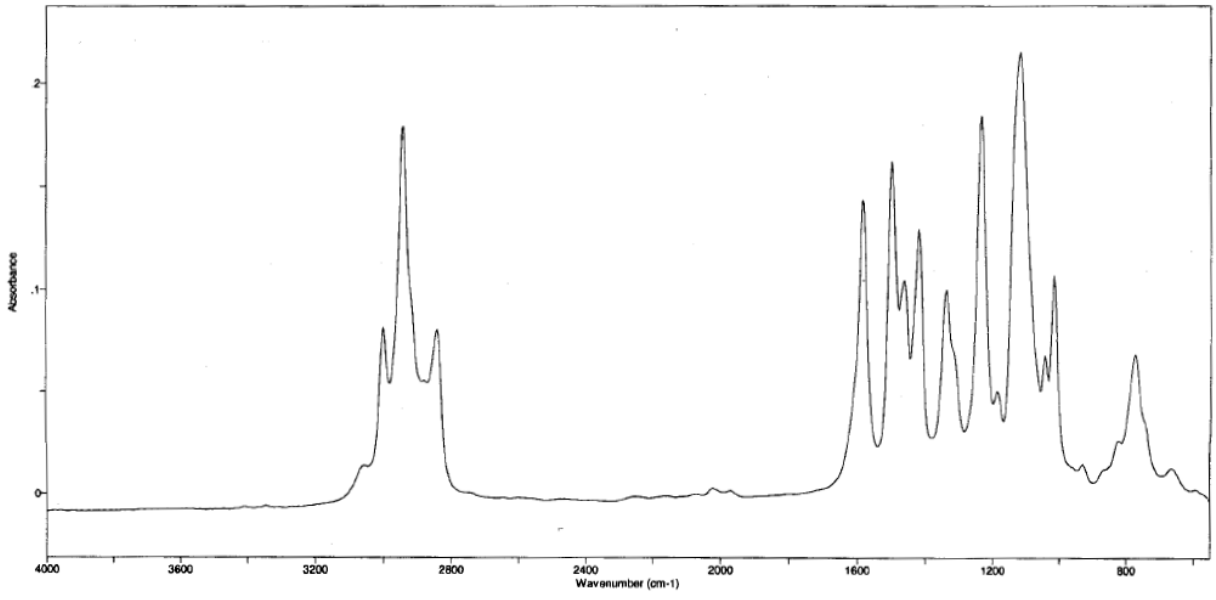
32 scans, 4 cm-1 resolution



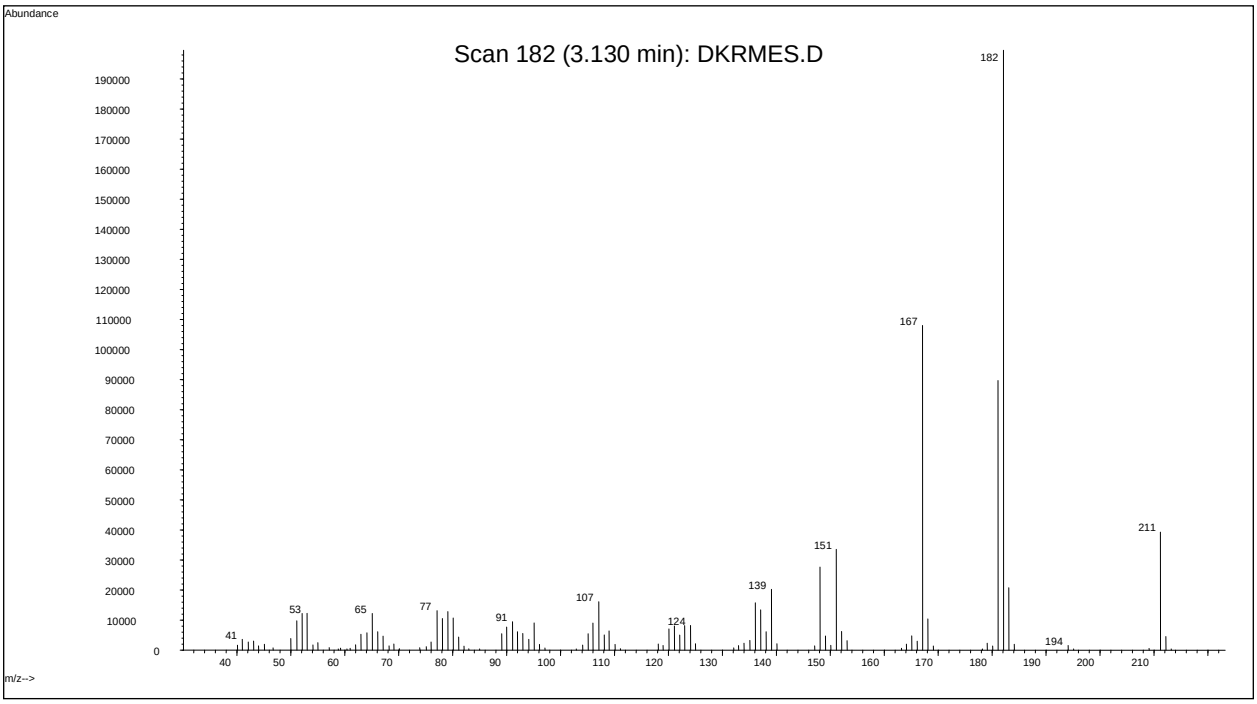
FTIR (Sample on single bounce ATR): Mescaline Hydrochloride
24 scans, 4cm^{-1} resolution

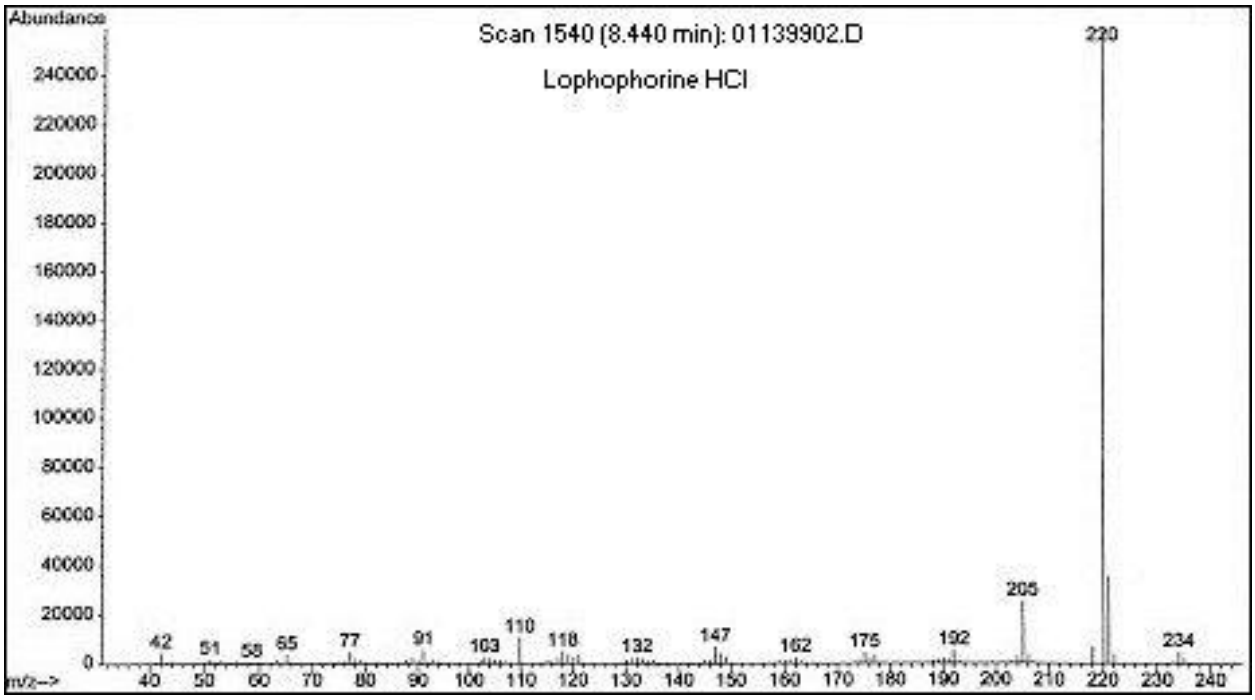


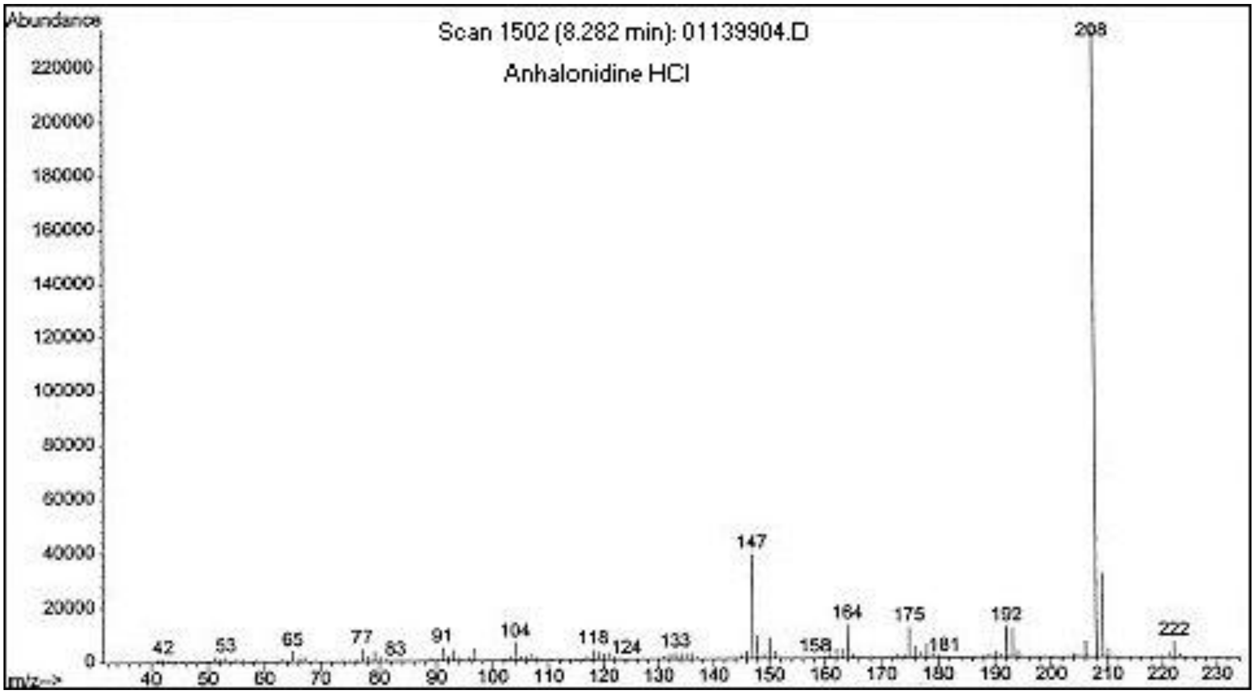
FTIR (VAPOR PHASE): Mescaline
4 scans, 8cm^{-1} resolution, Flow Cell Temperature 260°C

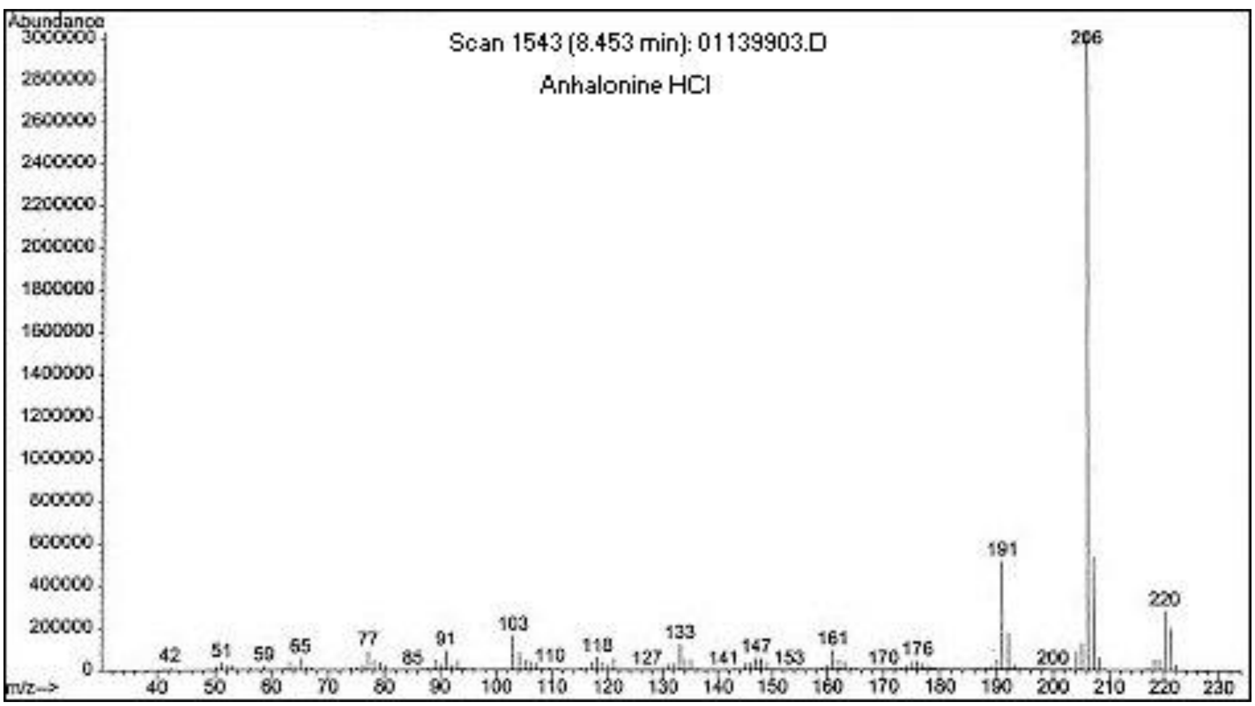


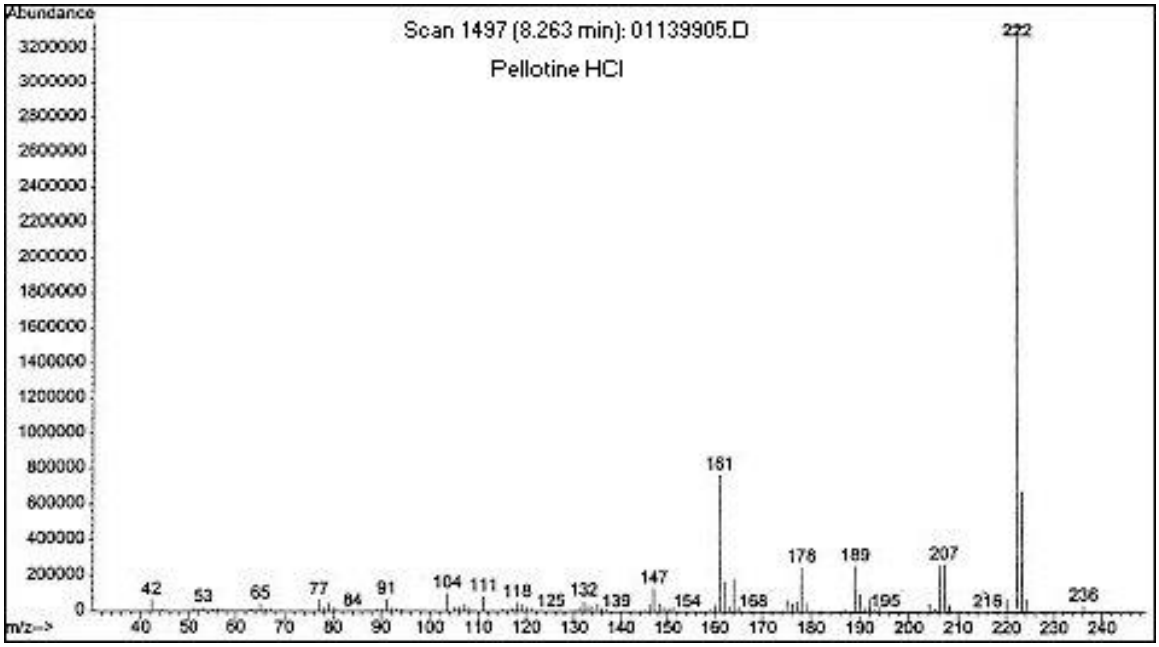
MS: Mescaline





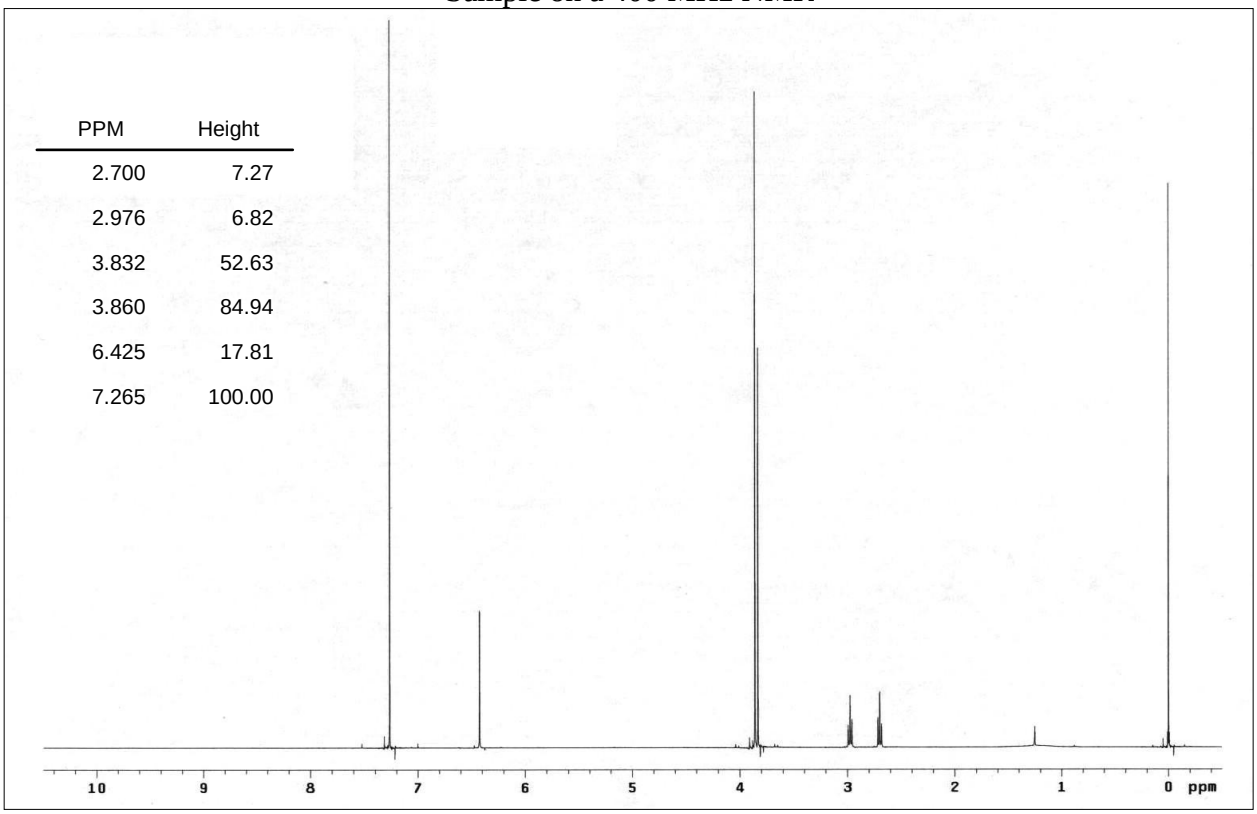

Abundance
Scan 1502 (8.282 min): 01139904.D
Anhalonidine HCl
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
208
147
164
175
192
222
42
53
65
77
83
91
104
118
124
133
158
181
m/z-->
40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230


Abundance
Scan 1543 (8.453 min): 01139903.D
Anhalonine HCl
3000000
2800000
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
206
191
220
42
51
59
65
77
85
91
103
110
118
127
133
141
147
153
161
170
176
200
m/z-->
40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230



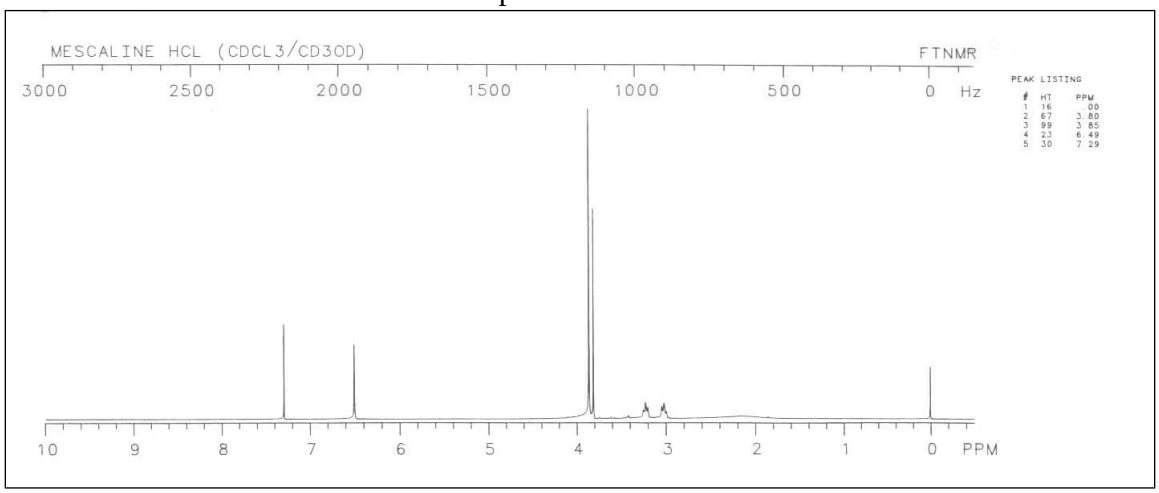
Mass spectral data for lophophorine, anhalonidine, anhalonine, pellotine obtained from Maloney, D.C., Microgram 2001; 34(8) 205

NMR (PROTON): Mescaline Base in $CDCl_3$
Sample on a 400 MHz NMR



PPM	Height
2.700	7.27
2.976	6.82
3.832	52.63
3.860	84.94
6.425	17.81
7.265	100.00

NMR (PROTON): Mescaline HCl in $CDCl_3/CD_3OD$
Sample on a FTNMR



NMR data for mescaline HCl obtained from Mills III, T. and Roberson, J. C., *Instrumental Data for Drug Analysis*, 2nd Edition, Volume 2, Elsevier 1987, p.1359.