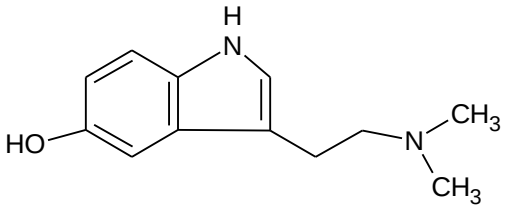
BUFOTENINE

Latest Revision: August 16, 2005



1. SYNONYMS

CFR: Bufotenine

CAS #: Base: 487-93-4

Other Names: N,N-dimethylserotonin, 5-hydroxy-N,N-dimethyltryptamine, mappine

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (^{o}C)
Base	$C_{12}H_{16}N_2O$	204.3	146-147
Oxalate Hydrate	$C_{12}H_{16}N_2O \cdot C_2H_4O_2 \cdot H_2O$	312.32	97-99

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	***	***	SS	***	FS	I
Oxalate Hydrate	VSS	SS	I	I	FS	VS

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

Note: Bufotenine is soluble in dilute acids and alkalis.

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Marquis	Green-brown

Van Urks	Violet to dark purple

3.2. THIN LAYER CHROMATOGRAPHY

Visualization

Van Urk's reagent

COMPOUND	RELATIVE R_F SYSTEM TLC 4
bufotenine	1.0
dimethyltryptamine	1.3

3.3. GAS CHROMATOGRAPHY

Method BUF-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 12 m x 0.2 mm x 0.33µm film thickness

Carrier gas: Hydrogen at 1.0 mL/min

Temperatures: Injector: 270°C
Detector: 280°C
Oven program:
1) 165°C initial temperature for 1.0 min
2) Ramp to 280°C at 30°C/min
3) Hold final temperature for 3.8 min

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

Samples are to be dissolved in 4:1 chloroform: methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
dimethylsulfone	0.15	caffeine	0.78
methamphetamine	0.24	lidocaine	0.83
ephedrine	0.35	procaine	0.95

3,4-MDA	0.45	psilocin	0.95
3,4-MDMA	0.50	**bufotenine**	**1.00 (3.85 min)**
benzocaine	0.52	cocaine	1.10
acetaminophen	0.63	tetracosane	1.20
phenacetin	0.64	heroin	2.84
dimethyltryptamine	0.73	quinine	1.67

3.5. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method BUF-LCS1

Instrument: High performance liquid chromatograph equipped with diode array

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

Detector: UV, 210 nm

Flow: 1.0 mL/min

Injection Volume: 5µL

Buffer: 0.1% (v/v) Trifluoroacetic acid in water

Mobile Phase: 80% Buffer : 20% Acetonitrile

Samples are to be dissolved in methanol and filtered with a 0.45 micron filter.

COMPOUND	RRT	COMPOUND	RRT
psilocybin	0.71	psilocin	1.31
bufotenine	**1.00 (2.32 min)**		

3.4. CAPILLARY ELECTROPHORESIS[4]

Method BUF-CES1

Injection Solvent:
3.75 mM sodium phosphate monobasic pH = 3.2.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of bufotenine oxalate hydrate at 0.5 mg/mL in methanol.

Sonicate the methanol solution for 5 min. Take 1 mL of the methanol solution and add it to 11 mL of injection solvent. Filter with a 0.45 µm or smaller filter.

Sample Preparation:
Weight out an appropriate amount of sample into an Erlenmeyer flask. Add enough methanol to cover the sample material and sonicate the sample solution for 1 hour. Filter 2-3 mL of the methanol solution with a 0.45 µm or smaller filter. Take 1 mL of the filtered methanol solution and add it to 11 mL of injection solvent. Filter with a 0.45 µm or smaller filter. Target the sample concentration to be approximately the sample concentration of the sample if possible.

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 pH to 1.8
Detector:	UV, 222 nm
Voltage:	10 kV
Temperature:	15°C aired 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:	Flush with 0.1 N NaOH for 1 min Flush with water for 1 min Flush with Microsolv CElixer A for 1 min Flush MicroSolv DEA custom chiral pH to 1.8 for 2 min

COMPOUND	RMT	COMPOUND	RMT
psilocin	0.93	psilocybin	1.94
bufotenine	**1.00 (6.64 min)**		

4. SEPARATION TECHNIQUES

Bufotenine is an indole alkaloid obtained from the seeds and leaves of *Piptadenia peregrine* and *P. macrocarpa*. It has also been isolated from the skin glands of toads (Bufo species). The base is typically seen as white crystalline powder and the oxalate hydrate as a lavender to brown powder. The oxalate hydrate is slightly soluble in chloroform and can be separated from chloroform insoluble material for analysis.

5. QUANTITATIVE DATA

N/A

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)
Aqueous Acid	278

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

7. REFERENCES

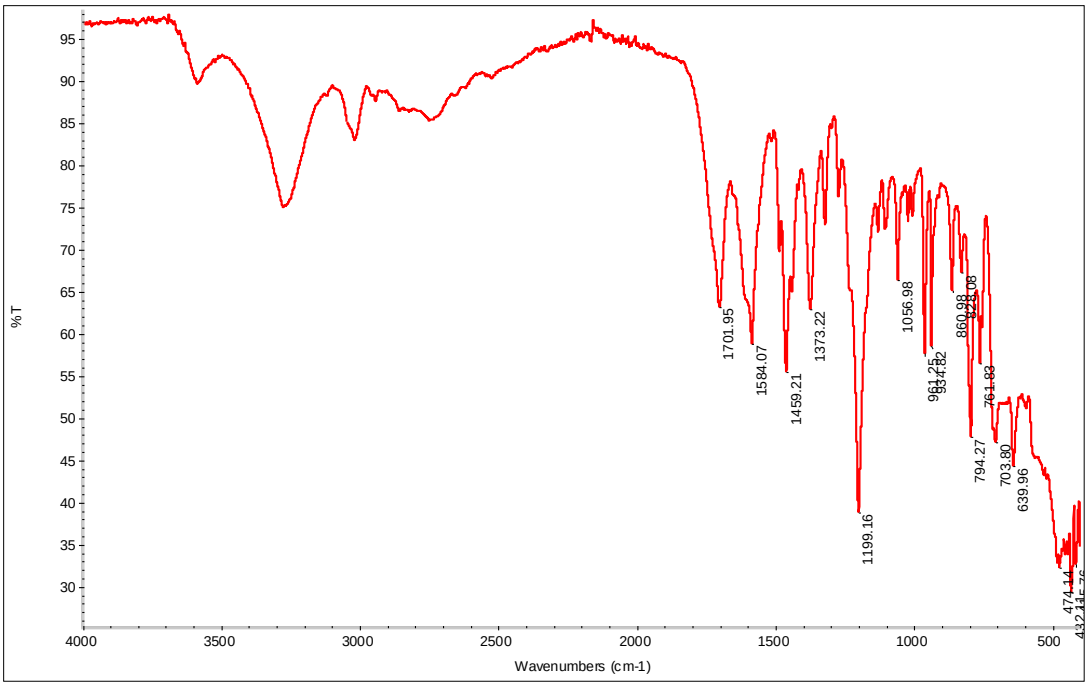
1) Moffat, A., Osselton, M.D., Widdop, B., *Clarke's Analysis of Drugs and Poisons, 3rd Edition*, The Pharmaceutical Press, 2004.
2) Mill, T., Robertson, J.C., *Instrumental Data for Drug Analysis., 2nd Edition,* Elsevier Science Publishing Co, 1987.
3) Budavari, S., *The Merck Index, 12th Edition*, Merck and Co., Inc., 1996.
4) Lurie, I.S., *et al*., Electrophoresis 2004, 25, 1580-1591.
*** = data not available

8. ADDITIONAL RESOURCES

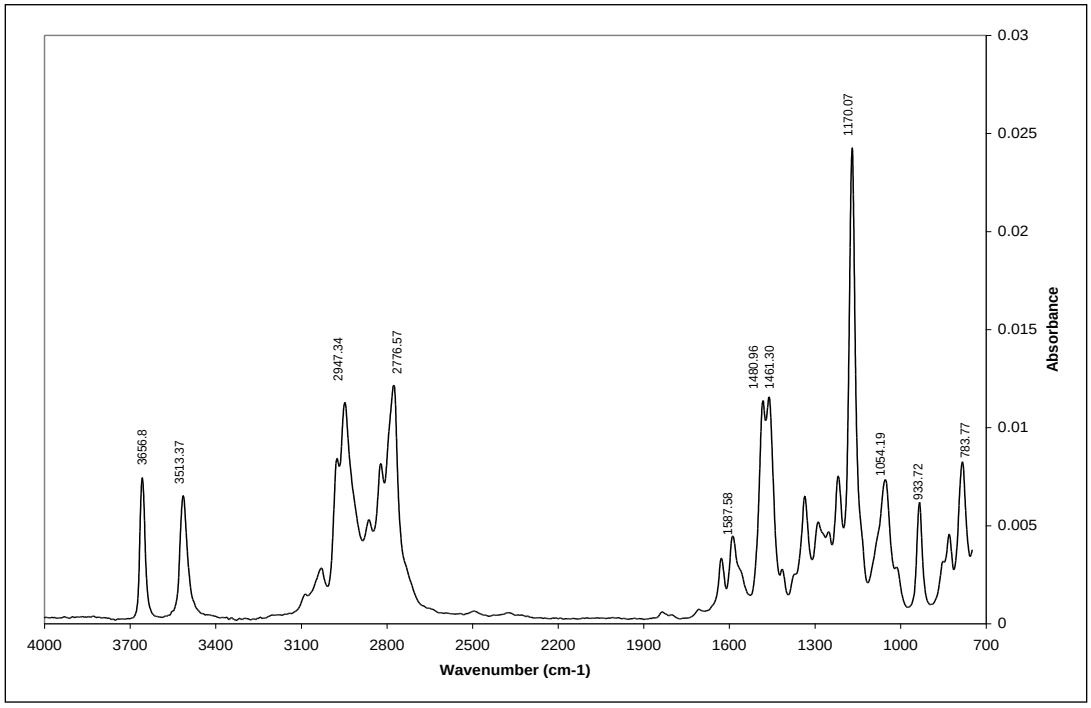
Forendex

Wikipedia

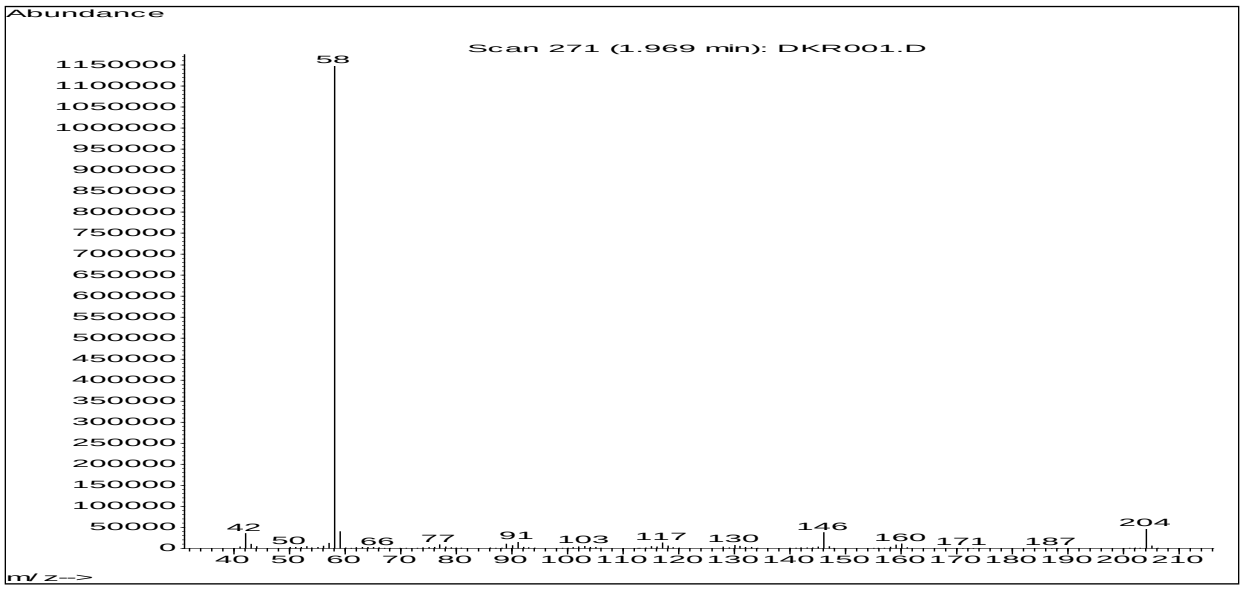
FTIR (ATR, single bounce): Bufotenine Oxalate Hydrate
32 scans, 4 cm^{-1} resolution



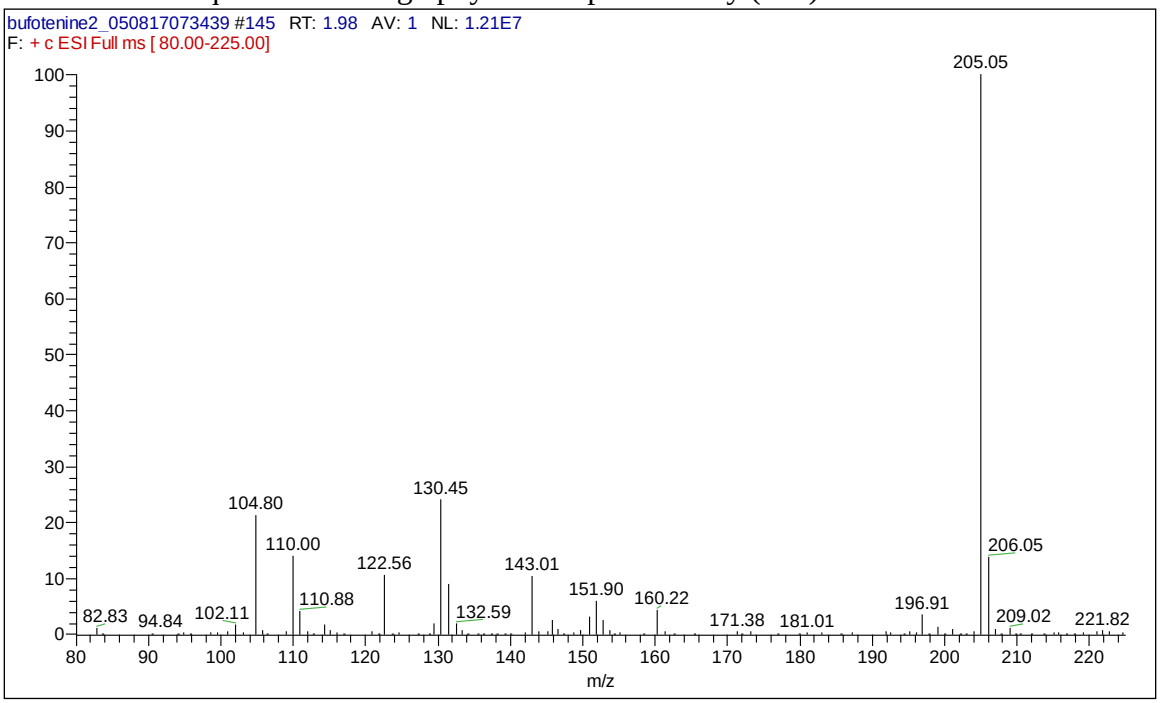
Vapor Phase FTIR: Bufotenine
4 scans, 8 cm^{-1} resolution, Flow Cell Temperature 260°C



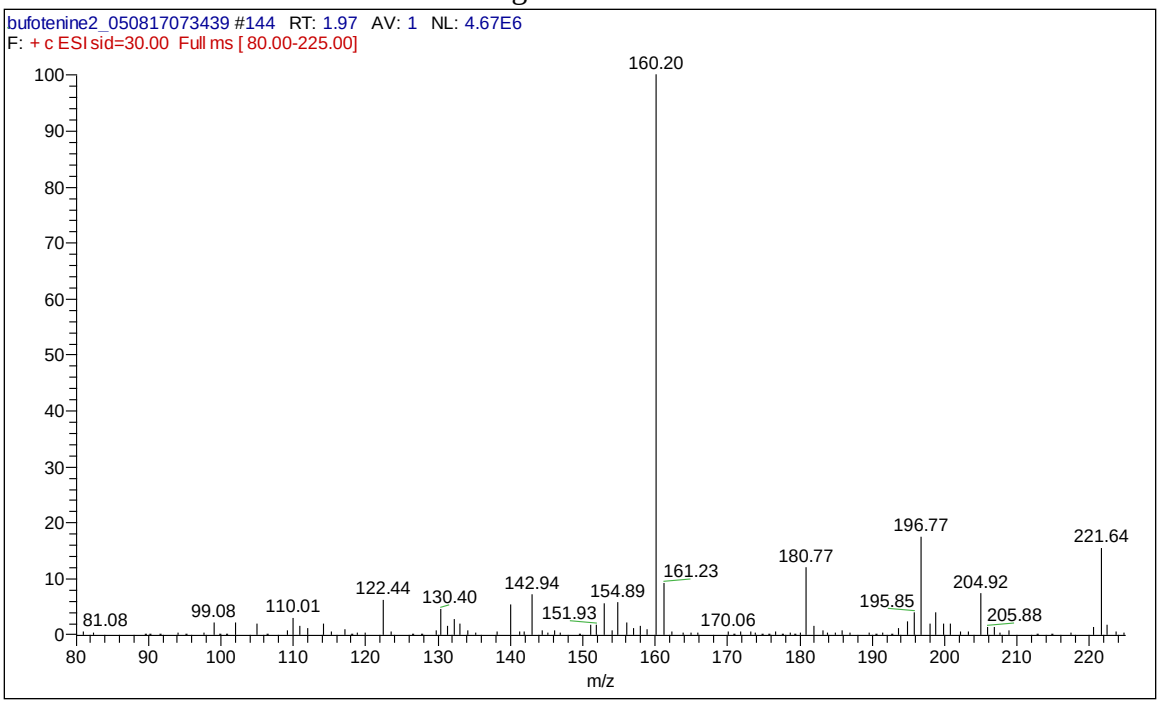
MS: Bufotenine



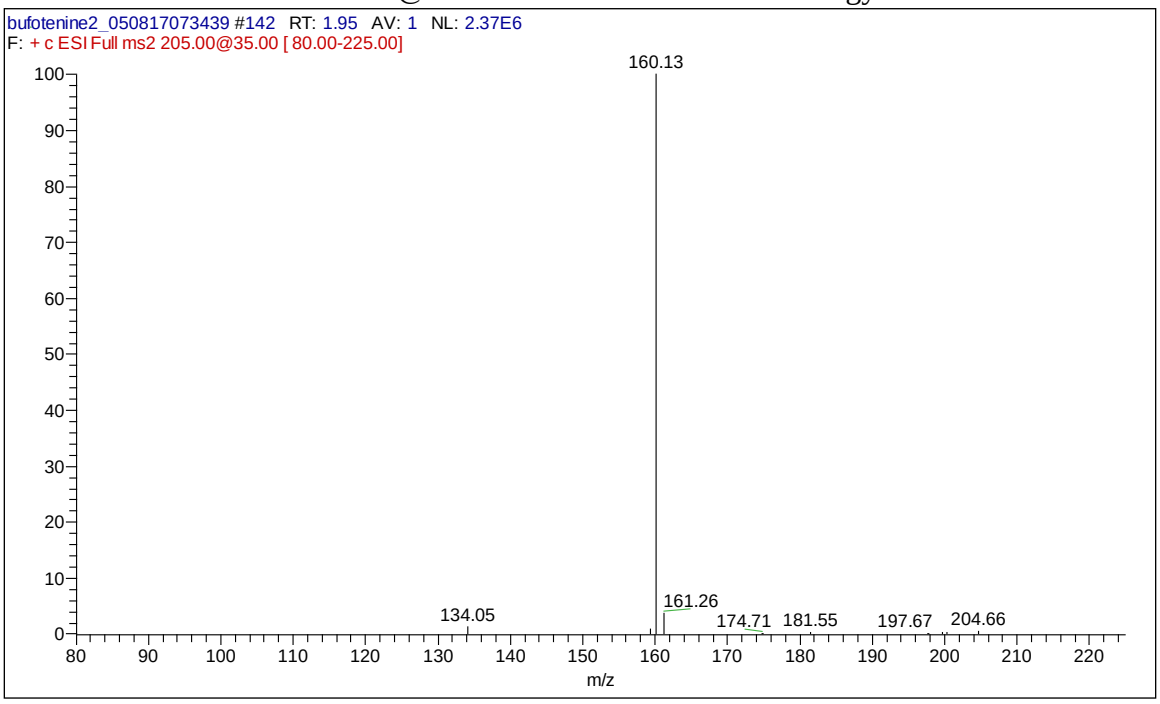
Liquid Chromatography/Mass Spectrometry (ESI): Bufotenine



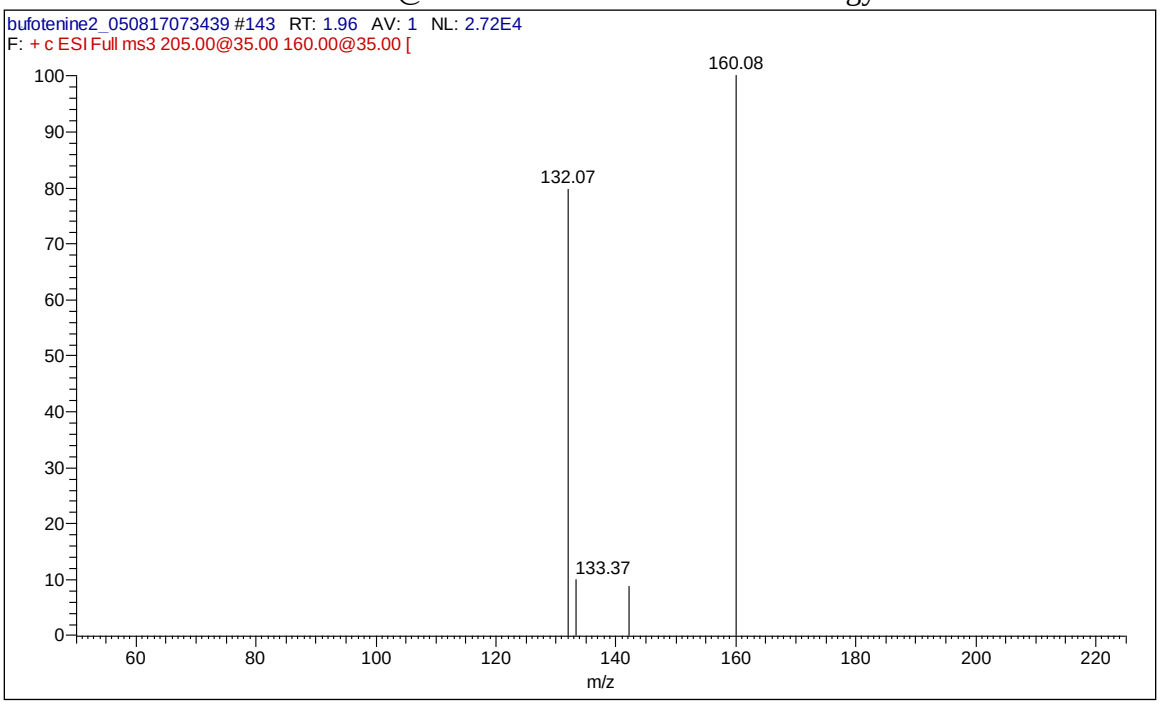
Liquid Chromatography/Mass Spectrometry (ESI): Bufotenine
Source Fragmentation on at 30V



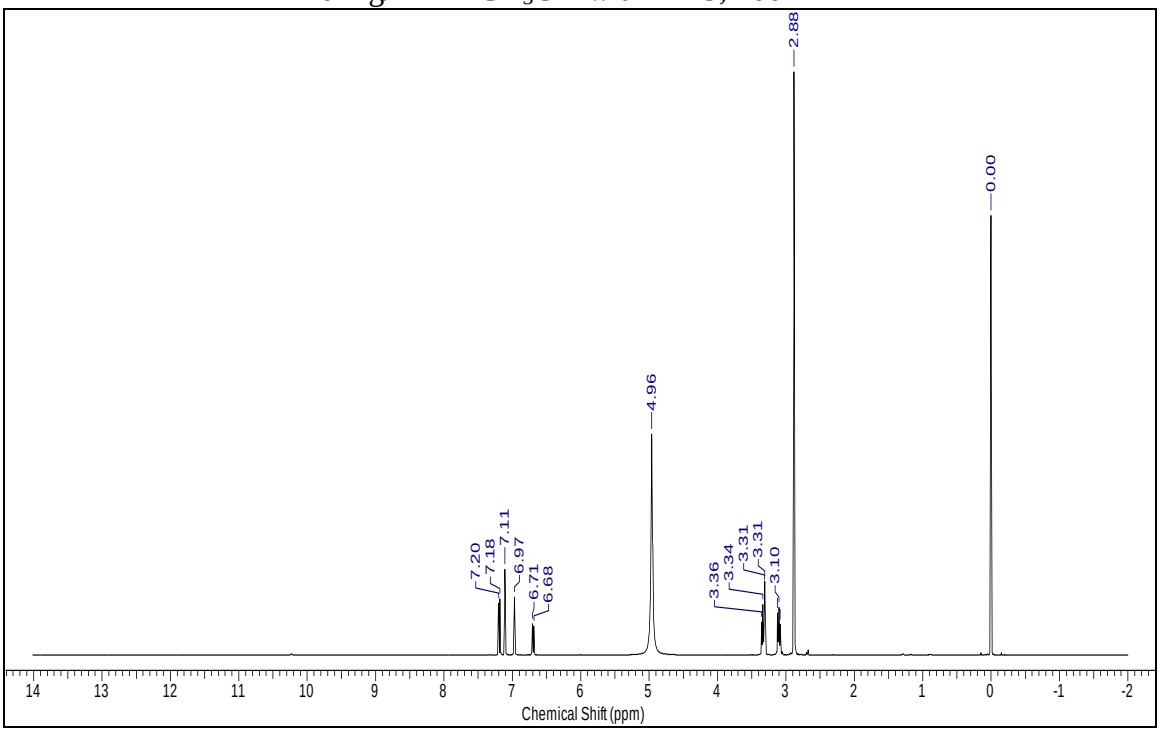
Liquid Chromatography/Mass Spectrometry (ESI): Bufotenine MS/MS
205 m/z @ 35% Normalized Collision Energy



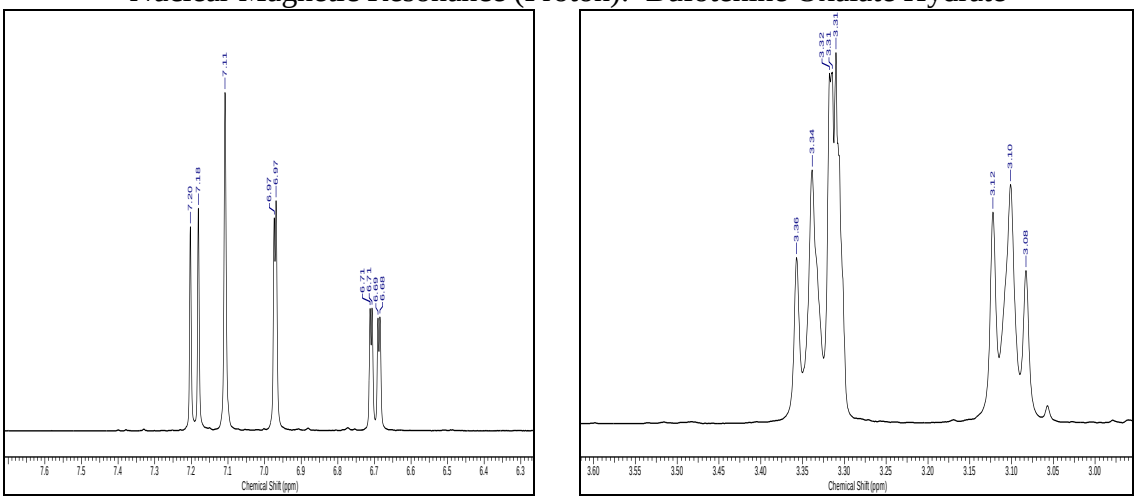
Liquid Chromatography/Mass Spectrometry (ESI): Bufotenine MS^3
205 m/z @ 35% Normalized Collision Energy
160 m/z @ 35% Normalized Collision Energy



NMR (Proton): Bufotenine Oxalate Hydrate
20 mg/mL in CD_3OD with TMS, 400 MHz



Nuclear Magnetic Resonance (Proton): Bufotenine Oxalate Hydrate



NMR (Carbon): Bufotenine Oxalate Hydrate
20 mg/mL in CD_3OD with TMS, 400 MHz