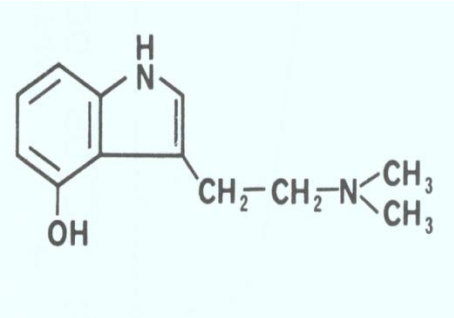
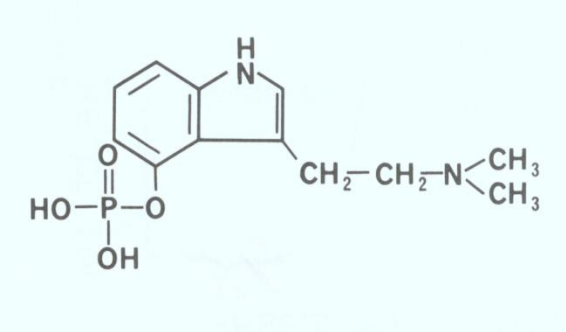
PSILOCIN **PSILOCYBIN** **Latest Revision: June 27, 2005**





1. SYNONYMS

Psilocin	***CFR:***	Psilocin, § 1380.11 Schedule 1 (d) Hallucinogenic Substances (26) Psilocin 7438
	CAS #:	520-53-6
	Other names:	Psilocyn 4-Hydroxy-*N*,*N*-dimethyltryptamine 3-[2-(Dimethylamino)ethyl]-1*H*-indol-4-ol; 4-Hydroxydimethyltryptamine
Psilocybin	***CFR:***	Psilocybin, § 1380.11 Schedule 1 (d) Hallucinogenic Substances (25) Psilocin 7437
	CAS #:	520-52-5
	Other names:	Psilocybine 4-Phosphoryloxy-*N*,*N*-dimethyltrypamine Indocybin CY-39

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Psilocin	$C_{12}H_{16}N_2O$	204.3	173-176
Psilocybin	$C_{12}H_{17}N_2O_4P$	284.3	185-195

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Psilocin	S	S	S	S	S	SS
Psilocybin	SS	I	SS	I	SS	S

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

COMPOUND	REAGENT	COLOR PRODUCED
Psilocin	Marquis	Greenish-brown
	Froehde's	Greenish-blue → Yellow
Psilocybin	Marquis	Dull Orange
	Froehde's	Grey-blue → Green →Yellow

3.2. CRYSTAL TESTS (Ref Clarke, Vol.1)

COMPOUND	REAGENT	CRYSTALS FORMED
Psilocin	Gold Cyanide Solution	Branching Needles, Best viewed under Polarized Light
	Trinitrobenzoic Acid	Bunches of Needles
Psilocybin		None Found

3.3. THIN-LAYER CHROMATOGRAPHY

Visualization

Van Urk Reagent

COMPOUND	System TLC5

Psilocybin (grey-violet)	0.05
Mescaline	0.22
Bufotenine	0.32
Psilocin (faint blue)	0.34

3.4. GAS CHROMATOGRAPHY

Method PSI-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 12 m x 0.2 mm 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°2C/min
3) Hold final temperature for 4.0 min

Injection Parameters: Split Ration = 60:1, 3 µL injected

Samples are dissolved in methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.16	cocaine	1.28
methamphetamine	0.17	tetracosane	1.47
psilocin	**1.00 (4.44 min)**	heroin	1.86

Psilocybin, when heated, gets dephosphorylated and coverts into Psilocin. Therefore, Gas Chromatography is not useful in screening for Psilocybin.

3.5. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PSI-LCS

Instrument: High performance liquid chromatograph equipped with diode array

Column:	Waters SymmetryShield RP18 (4.5 mm x 150mm, 3.5µm)
Detector:	UV, 210 nm
Flow:	1.0 mL/min
Injection Volume:	5.0 µL
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 methanol and filtered with a 0.45-micron filter.

COMPOUND	RRT	COMPOUND	RRT
ephedrine	0.68	**psilocin**	**1.00 (6.08 min)**
pseudoephedrine	0.75	methamphetamine	1.11
amphetamine	0.92	**psilocybin**	**1.25 (7.61 min**)

4. SEPARATION TECHNIQUES

Two major factors complicate the identification of psilocin and psilocybin. First, chromatographic problems arise due to large amounts of alkaloids and sugars present. Second, the dephosphorylation of psilocybin due to its hydrolytic instability inhibits conventional solvent purification.

The identification of psilocin and psilocybin in mushrooms can be accomplished by: 1) drying the mushroom material 2) adding methanol and grinding the sample 3) filtering out the leftover particles 4) adding acetone to precipitate out the sugars 5) and then filtering and drying the sample. The sample can now be used for color tests, micro-crystal tests, HPLC, and TLC.

The TLC analysis can be further used to isolate the individual compounds. Once isolated, qualitative analysis such as IR and NMR can be performed. In addition, a GC-MS can be obtained from Psilocybin by derivatizing the compound with trimethylchlorosilane (TMCS) and N,O-bis(trimthylsilyl)trifluoroacetannide (BSTFA).

5. QUANTITATIVE PROCEDURES

N/A

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

FORM	SOLVENT	ABSORBANCE

		MAXIMA (NM)
Psilocin	Aqueous Acid	266, 283, 292
	Aqueous Alkali	270, 293
Psilocybin	Aqueous Acid	268
	Aqueous Alkali	269, 282, 292

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

7. REFERENCES

Budavari, S., *The Merck Index, 11th Edition*, Merck and Co., Inc., 1989.

Clarke, E.G.C., *Isolation and Identification of Drugs, Volume 1*, The Pharmaceutical Press, 1975.

Miller, D., Microgram 1972, 1(5):4-10.

Mills, T., Roberson, J., *Instrumental Data for Drug Analysis, 2nd Edition, Volume 3*, Elsevier, 1987.

Moffat, A., Osselton, M., Widdop, B., *Clarke's Analysis of Drugs and Poisons, 3rd Edition, Volume 2*, The Pharmaceutical Press, 2004.

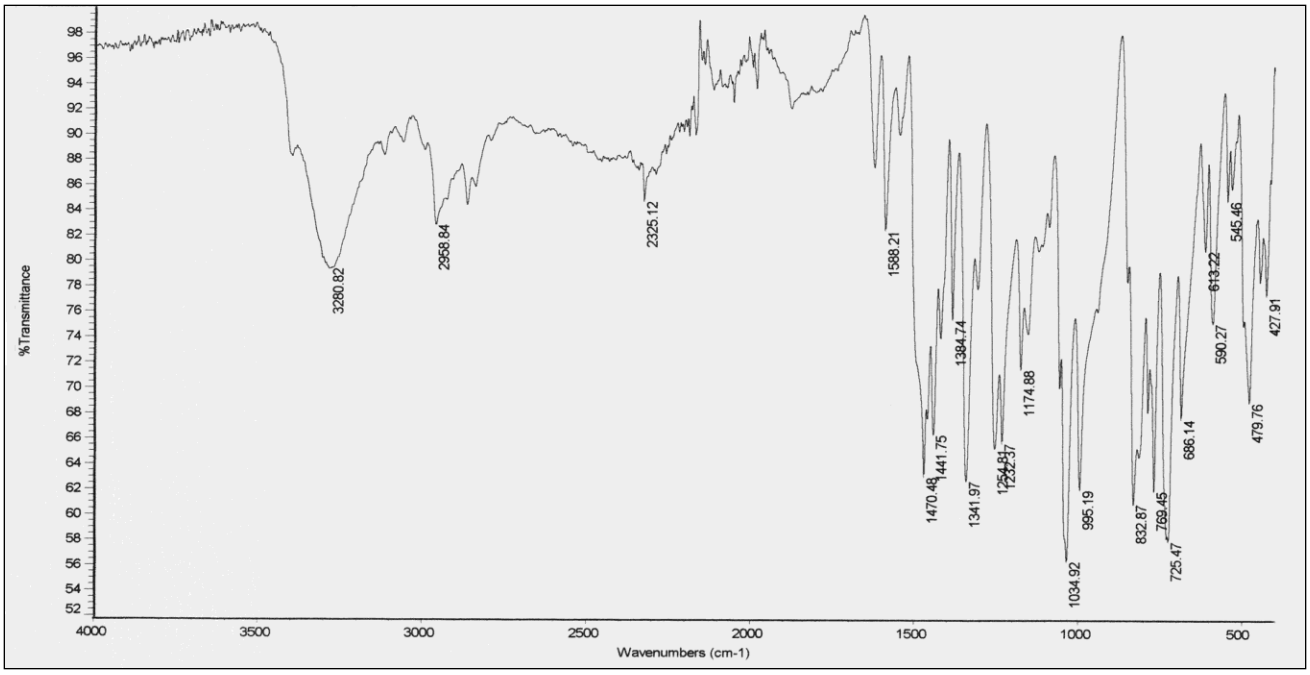
Timmons, J., Microgram 1984, 2(17):10-12.

8. ADDITIONAL RESOURCES

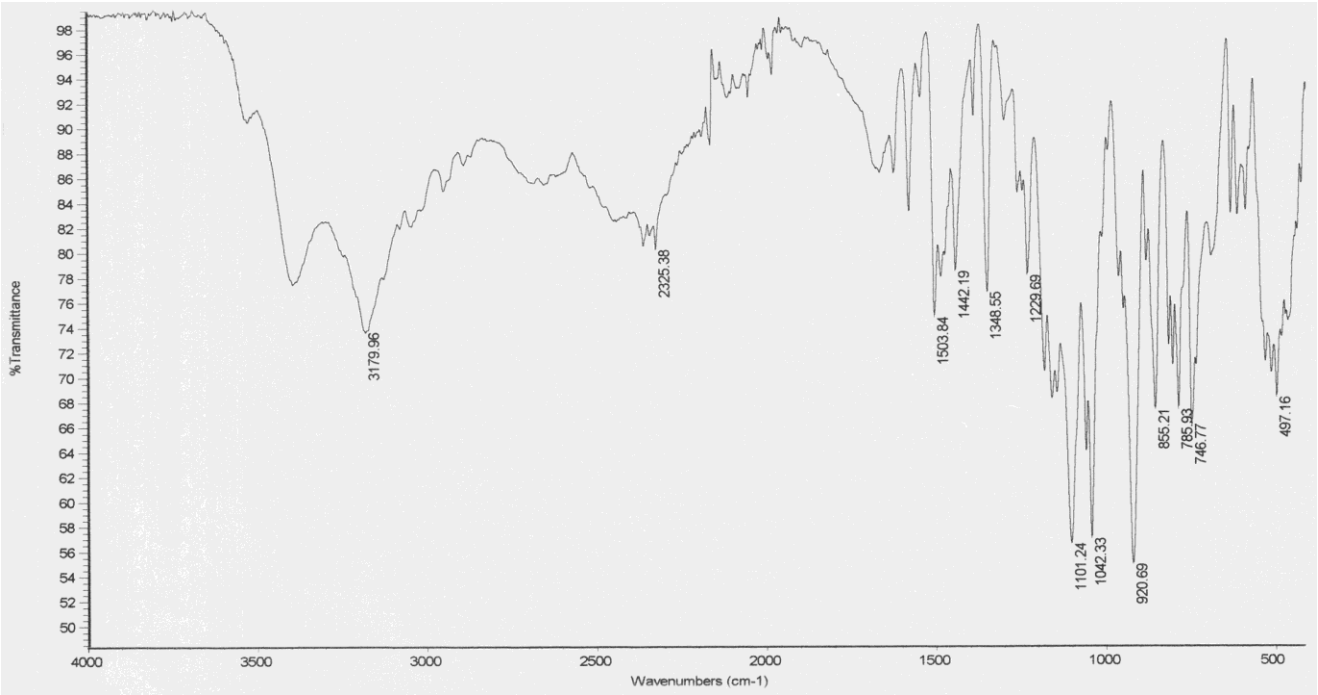
Forendex (Psilocin and Psilocybin)

Wikipedia (Psilocin and Psilocybin)

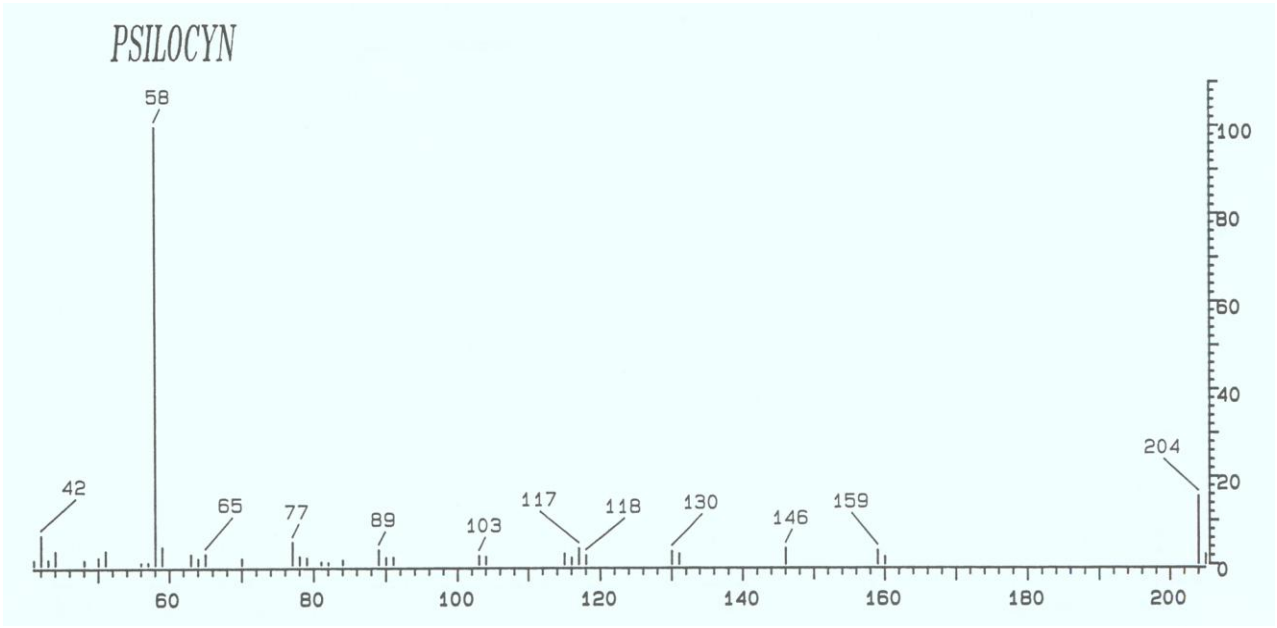
FTIR (single bounce ATR): Psilocin
32 scans, 4cm^{-1} resolution



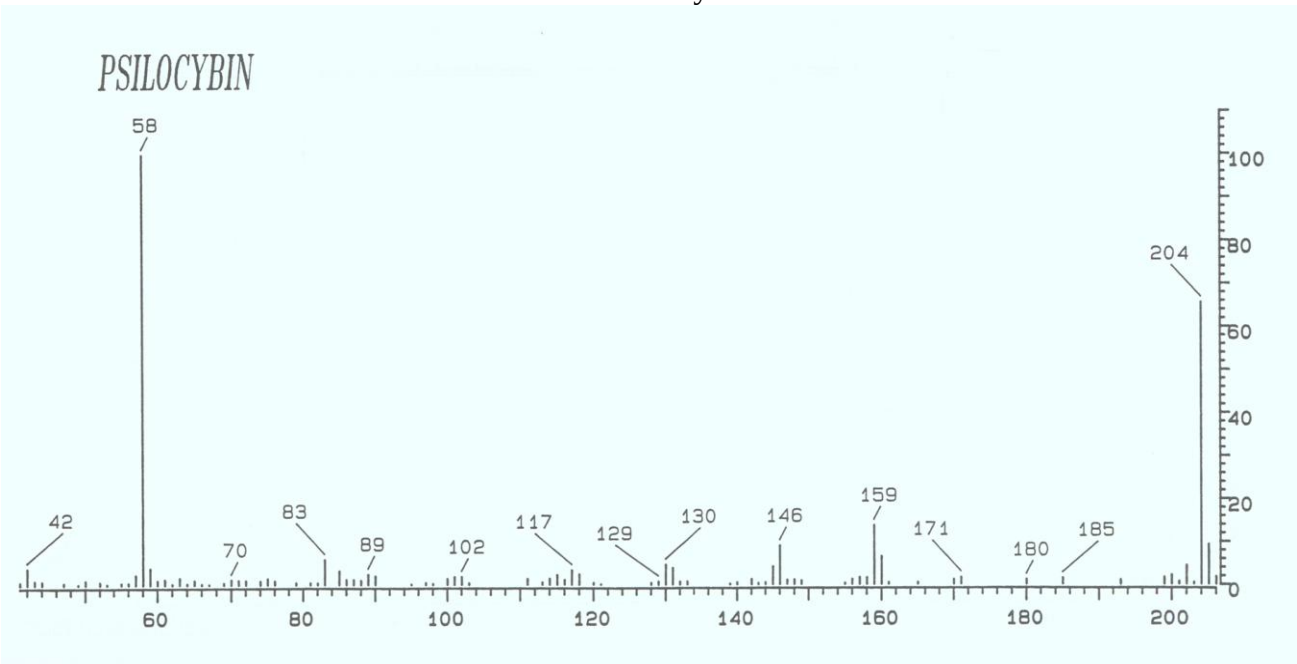
FTIR (single bounce ATR): Psilocybin
32 scans, 4cm^{-1} resolution



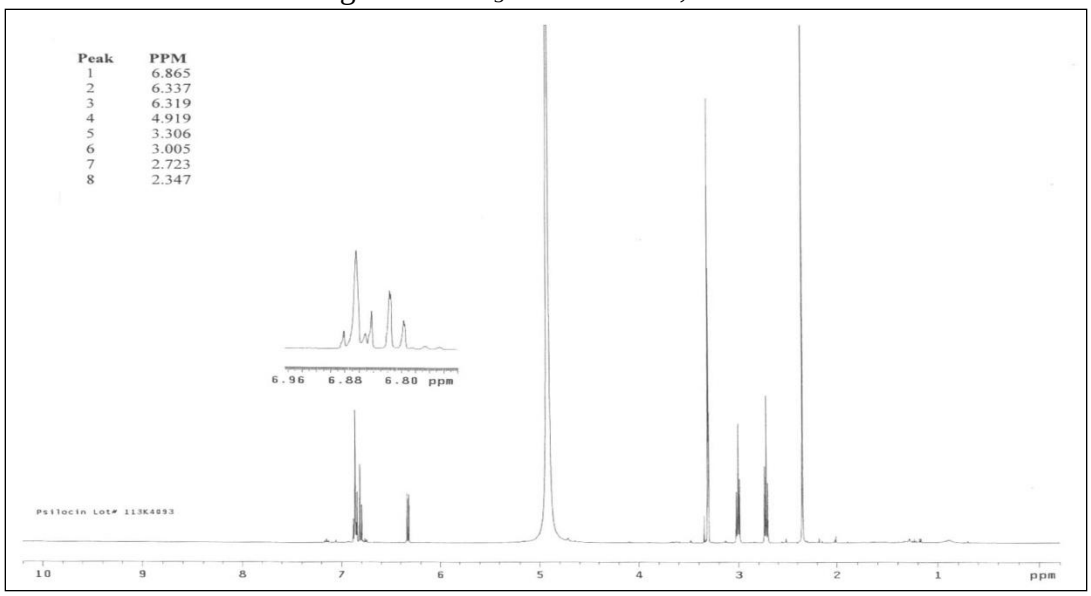
MS: Psilocin



MS: Psilocybin



NMR (PROTON): Psilocin
10 mg/mL in CD_3OD with TSP, 400 MHz



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