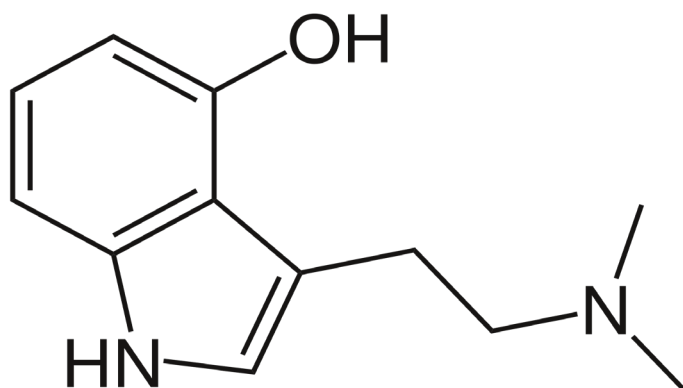


Psilocin In Urine For GC Or GC/MS Confirmations Using: 200 mg Clean Screen® Extraction Column



UCT Part Numbers

ZSDAU020
Clean Screen® DAU
10 mL, 200 mg Column

Or

ZCDAU020
Clean Screen® DAU
10 mL, 200 mg Column
with CLEAN-THRU® Tips

Procedure:

1. Prepare Sample

- To 5 mL of urine add internal standard and 2 mL of 100 mM phosphate buffer (pH 6.0).
- Mix/vortex.
- Add 12,500 to 25,000 units of β -Glucuronidase, Mix/Vortex.
- Place the sample into a water bath at 45°C for 90 minutes.
- Remove from the bath and allow to cool.
- Centrifuge at 3,000 rpm for 10 min.
- Use the clear filtrate (discard the plug) for SPE.

2. Condition Clean Screen® Extraction Column

- 1 x 3 mL CH₃OH.
- 1 x 3 mL D.I. H₂O.
- 1 x 1 mL 100 mM phosphate buffer (pH 6.0).

Note: Aspirate at < 3 inches Hg to prevent sorbent drying.

3. Apply Sample

- Load at 1 mL/minute.

4. Wash Column

- 1 x 3 mL D.I. H₂O.
- 1 x 2 mL 20% Acetonitrile in water.
- 1 x 1 mL 100 mM Acetic Acid.
- Dry column (3 minutes at > 10 inches Hg).
- 1 x 2 mL Hexane.
- 1 x 3 mL Hexane/ Ethyl Acetate (50:50).
- 1 x 3 mL CH₃OH.
- Dry Column (3 min at > 10 inches Hg).

5. Elute Psilocin

- 1 x 3 mL Dichloromethane/ Isopropanol/Ammonium Hydroxide (78:20:2).
- Collect eluant at 1 mL /min.

NOTE: Prepare elution solvent daily.

6. Dry Eluate

- Evaporate to dryness at < 35°C.

7. Derivative

- Add 50 μ L of ethyl acetate. Vortex mix.
- Add 50 μ L of MSTFA*
- React for 30 minutes at 70°C.
- Remove from heat.

NOTE: Do not evaporate MSTFA solution.

8. Quantitate

- Inject 1 to 2 μ L onto chromatograph.
- Monitor the following ions (Mass Selective Detection):

Compound	Primary**	Secondary	Tertiary	Cerilliant #
PSILOCIN – TMS	290	348	73 (291)	P-048
*PSILOCIN-D10-TMS	300	358	83(301)	P-049

* Part #SMSTFA-0-1, 10, 25, 100

** Quantitation Ion



GC Conditions	
System	HP Model 5890 GC with a 5970 MSD
Column	DB5 (25 m x 0.32 mm ID x 0.17 µm Film Thickness)
Carrier Gas	Helium (5 psi head pressure)
Injection Size	1 µL (Splitless Mode)
Injection Temperature	275°C
Detector Temperature	300 °C
Temperature Program	70° C hold 1 min then ramp to 240 °C at 20 °C/min hold for 2 minutes

References:

[1] The Detection of Psilocin in Human Urine Grieshaber A, Moore K, Levine B and Smith M Presented at the TRI-SERVICES Meeting Nov 1999

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