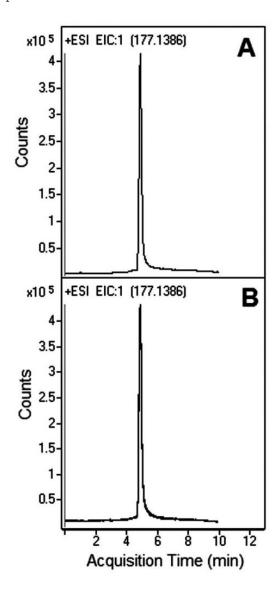


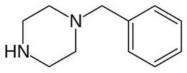
Analysis in Hair Samples using LCMS

An analytical protocol was developed for analysis of Benzylpiperazine in human hair samples. The elucidation of the chromatographic method was challenging due to the polar nature of BZP. The limits of detection / quantification for this method were determined to be 0.05 ng / mg for Benzylpiperazine in hair samples.

The method was found to be linear from 0.1 – 10 ng/mg (r2 > 0.999). Recovery of Benzylpiperazine was found to be greater than 95%. Matrix effects were determined to be < 6%.

The concentration of Benzylpiperazine in spiked samples of hair was determined in range from 1.2 - 1.5 ng/mg. The procedure after validation will be useful for laboratories performing routine analysis of drugs of abuse.





Benzylpiperazine

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Peaks:

A: Benzylpiperazine 177.1386 m/z [M+H]+ B: 5 injections of the sample

Method Conditions

Column: Cogent Diamond Hydride[™], 4μm, 100Å

Catalog No.: 70000-15P-2 **Dimensions:** 2.1 x 150mm

Mobile Phase:

A: DI Water / 0.1% Formic Acid (v/v)
B: Acetonitrile / 0.1% Formic Acid (v/v)

Gradient:

| Time (minutes) | %B |
|----------------|----|
| 0 | 80 |
| 3 | 30 |
| 6 | 30 |
| 10 | 80 |

Flow rate: 0.4 mL/minute

Detection: ESI - POS - Agilent 6210 MSD TOF Mass Spectrometer

Injection vol.: 1µL

Sample Preparation: Extraction (SPE) was performed on cartridge I (Clean Screen $Xcel^{™}$ purchased from UCT Bristol, PA, USA), preconditioned with 3mL of Methanol, 3 mL of DI Water, and 1 mL of pH 6 buffer prior to sample loading. 10 mg samples of hair (controls, and spiked test samples) were digested in 1 mL of 1 M Sodium Hydroxide for 1 hour at 70°C. The samples were cooled, and 100 µL of Acetic Acid (Glacial) was added.

Each solution was adjusted to pH 6 with 0.1 mM Ammonia and applied to the SPE column. After loading the samples, each sorbent was washed with DI Water, Acetic Acid (0.1 M), and Methanol (3 mL of each, respectively). Each SPE Column was dried and eluted with 3 mL of Methylene Chloride / Isopropanol / Ammonium Hydroxide (78:20:2). After elution, solvents were evaporated and 200 μ L of Mobile Phase was added. The samples were used for analysis by LCMS.



Attachment

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