OVERVIEW

Purpose
- Rapid analysis of synthetic phenethylamines (25I-NBOMe and 25B-NBOMe) in urine, saliva, and serum using LDTD-
  MS/MS

Method
- Standards, QC and sample preparation using 3 different matrices
- Liquid-Liquid extraction
- Deposit of small volume of the organic phase in a Lazer™ plate
- Ultra-Fast quantitative analysis using LDTD-MS/MS system

Results
- Excellent linearity over the calibration range (R² > 0.998)
- Accuracy ranging from 98.6 to 110.9%
- Precision ranging from 0.3 to 4.45%
- All samples are analyzed with a run time of 9 seconds using LDTD-MS/MS system

INTRODUCTION

Emerging synthetic drugs of abuse have been introduced on the illegal drug market and pose a challenge for analytical forensic toxicologists. These drugs continue to increase in numbers and slightly vary across different chemical classes, making them difficult to detect by current analytical methods. In November 2013, the United States Drug Enforcement Administration (DEA) issued a final order to place these two synthetic phenethylamines into the Controlled Substances Act. A comprehensive detection and quantification method for the analysis of these emerging drugs of abuse in biological matrices is needed. We developed an ultra-fast, high-throughput and cost effective analytical method for the detection of 25I-
NBOMe and 25B-NBOMe using the LDTD® coupled to a tandem mass spectrometry system. Method development was performed in urine, saliva (Oralasure® buffer) and serum matrices and the analysis time needed was 9 seconds per sample.

LDTD® Ionization Source:
The LDTD® uses a Laser Diode to produce and control heat on the sample support (Figure 1), which is a 96 well plate. The energy is then transferred through the sample holder. The sample gets dried and vaporized prior being carried by a gas in a corona discharge line. This type of ionization is characterized by a strong resistance to ionic suppression because of the absence of solvent. LDTD® ionization reduces sample-to-sample analysis time to 9 seconds and allows high throughput capabilities without carry over.

METHOD

Liquid-liquid extraction
- 50 μL standard, QC or patient specimen
- 20 μL internal standard (25I-NBOMe-D3, 50 ng/mL in MeOH)
- 200 μL buffer NaClO₂ 0.5M pH 10
- 200 μL hexane/ethyl acetate: 75:25 v/v
- Vortex and centrifuge at 1400 rpm for 2 minutes
- Transfer 6 μL of the organic layer in a Lazer™ plate
- Analyze after complete solvent evaporation

Instrumentation
- LDTD model 5600, Sciex Technologies
- TQPARP 5500 System, Sciex

LDTD Parameters
- Linear power pattern
- Increase laser power to 45 % in 6.0 s
- Hold for 2 seconds
- Decrease laser power to 0 %
- Carrier-gas flow: 3 L/min (Ar)

RESULTS:

Precision and Accuracy
As shown in following Tables 2, 3, 4, an in-run intra precision for 25I-NBOMe and 25B-NBOMe in different matrices between 0.30% and 4.45% and an accuracy ranging from 98.3% and 110.5%.

Liquid-Liquid extraction

<table>
<thead>
<tr>
<th>Substance</th>
<th>Amount (ng/mL)</th>
<th>Mean</th>
<th>RSD</th>
<th>Mean</th>
<th>RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzoylecgonine</td>
<td>8.11</td>
<td>8.50</td>
<td>4.80</td>
<td>10.54</td>
<td>0.68</td>
</tr>
<tr>
<td>Caffeine</td>
<td>0.91</td>
<td>1.03</td>
<td>1.23</td>
<td>1.97</td>
<td>0.97</td>
</tr>
<tr>
<td>Codeine</td>
<td>0.93</td>
<td>1.03</td>
<td>1.12</td>
<td>1.97</td>
<td>1.03</td>
</tr>
<tr>
<td>Norocodeine</td>
<td>1.03</td>
<td>1.03</td>
<td>1.03</td>
<td>1.03</td>
<td>1.03</td>
</tr>
</tbody>
</table>

Drugs Interferences
32 drugs, with a concentration of 1000 ng/mL, have been added to a 10 ng/mL quality control sample to analyze the interference between these drugs of the 2 phenethylamines. Table 5 shows the concentration results for both 25I-NBOMe and 25B-NBOMe drugs in the 3 different matrices while Table 6 shows the drugs used in the analysis.

CONCLUSIONS

- Fast extraction of 25I-NBOMe and 25B-NBOMe for serum, saliva, and urine samples
- High Selectivity, Sensitivity and Specificity using Tandem Mass Spectrometry
- Excellent linearity of the LDTD technology proven with human samples
- LDTD provides the High-Throughput analysis of sample extract in 9 seconds sample-to-sample without any carry over