K74 Analytical Method Development and Robustness Evaluation for Gas Chromatographic Analysis of Piperazine Designer Drugs

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After attending this presentation, attendees will have a better understanding of an analytical screening method for piperazine designer drugs by Gas Chromatography (GC) coupled to both Mass Spectrometry (MS), Flame Ionization Detector (FID), and Nitrogen Phosphorus Detector (NPD).

This presentation will impact the forensic science community by demonstrating thorough development of a GC analytical method for the detection of piperazine designer drugs. This method would provide crime laboratories with a more robust GC analytical method for screening piperazine-derived drugs and improve overall accuracy of base-type drug analysis.

Synthetic designer drugs are among the most commonly abused drugs on the market.[1] Designer drugs are compounds that are synthesized to simulate the structures and effects of illegal drugs of abuse. To evade the risk of being charged with using drugs, there has been a trend toward consumption of designer drugs instead of the illegal drugs of abuse. Synthetic piperazine-derived drugs are one of the many emerging types of synthetic designer drugs on the market. Piperazine-derived drugs are not only being consumed directly, but also being added to multiple different street drugs. Developing a method to analyze and detect piperazine compounds (i.e., BZP and TFMPP) will aid the screening for emerging synthetic piperazine drugs.

The objective of this research is to optimize the conditions and parameters for a method using simultaneous GC/MS/FID/NPD in order to screen for and detect piperazine-derived compounds. An NPD has higher sensitivity than an FID and a single quadruple MS system, but it is less specific than MS. Due to the amine group of the piperazines, NPD was used to quantitate, providing a lower level of detection than the MS. Additionally, the NPD is likely to exhibit less instrumental drift than the MS, making it a potentially better choice for quantification. In addition, piperazines may exhibit chemical reactivity in the GC sample pathway. A series of commonly used piperazine drugs were evaluated as part of a study to investigate the chemical reactivity. During the course of this research, the impact of the deactivation chemistry of the inlets and columns was evaluated, with results ranging from poor to excellent. Based on chromatographic probes, including tailing factor, the best combination of column and inlet liner was chosen.

The rapidly changing synthetic piperazine market and regulation of these compounds has created a need for developing a more reliable screening technique, which allows crime laboratories to handle various kinds of piperazine-derived compounds. This method increased the accuracy of the analysis by comparing the chemical reactivity in the GC pathway and increased the sensitivity and selectivity by coupling both MS and NPD. This complete investigation of the chromatographic variables as directed to both native piperazines, and their relevant metabolites, in both recreational drug samples and human urine will allow for a consolidated analytical methodology that is more efficient in a commercial laboratory.

Chart 1 Column Comparison Based on Average Tailing Factor Using a Restek-Based Deactivated Gooseneck Inlet Liner

Column	Avg. Tailing Factor
Rtx-5MS	1.728±0.127
Rxi-5SilMS	2.395±0.164
Rtx-5 Amine	2.021±0.062
Rtx-35	1.892±0.106
Rxi-35 SIlMS	2.094±0.27
Rtx-1301	2.611±0.13
Rxi-1301Sil	1.949±0.132

Chart 2 Gooseneck Inlet Liner Comparison Based on Average Tailing Factor Using a Rxi-35 SilMS Column

Liner	Avg. Tailing Factor
Base Deactivated	2.09±0.273
Siltek Deactivated	1.99±0.137

IP Deactivated	2.163±0.148
Sky Liner	2.652±0.215
Ultra Inert Liners	2.01±0.083

Reference:

1. Arbo, M.D., Bastos, M.L., Carmo, H.F. Piperazine Compounds as Drugs of Abuse. Drug Alcohol Depend. 2012, 122, 174–85.

Piperazine, GC/MS/NPD, Inertness