A204 Investigation Into Cyclohexanone as a Schiff-Base Derivatizing Agent for the Detection of Cathinones With Gas Chromatography/Mass Spectrometry

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After attending this presentation, attendees will have a better understanding of the use of cyclohexanone as a derivatizing reagent for cathinone compounds.

This presentation will impact the forensic science community by providing a simple method for screening and identification of the target cathinone analogues in illicit samples using cyclohexanone as a Schiff-base derivatizing agent and Gas Chromatography-Mass Spectrometry (GC/MS).

"Bath salts" have recently become an illicit drug problem across the United States. Originating in the United Kingdom, "bath salts" have spread nationwide and are currently being classified as illegal substances of abuse by most states. Illicit drug suppliers are trying to circumvent the control of these substances by altering the structural arrangement of the compounds creating new analogues. As a result, forensic scientists are scrambling to develop methods to detect and identify these illicit substances. Consequently, cyclohexanone was investigated as a derivatizing reagent for the detection by GC/MS of thirteen structurally similar *β*-keto-phenethylamine compounds which have evidence of abuse. Cathinone, cathine (norpseudoephedrine), methcathinone (ephedrone), 4'-methylmethcathinone (mephedrone), 4'-fluoromethcathinone (flephedrone), dimethylcathinone (diethylpropion), ephedrine, pseudoephedrine, norephedrine (phenylpropanolamine), methylpseudoephedrine, N-ethylcathinone (ethcathinone), 4'-methoxymethcathinone (methedrone), and 3,4-methylenedioxymethcathinone (methylone) were all examined using GC/MS. Derivatization of the cathinones by cyclohexanone via Schiff-base formation was simple and proved to be an efficient method. Using this method, it is possible to achieve separation and identify many structurally similar cathinone-related compounds in mixtures.

A GC/MS method for screening or confirming 13 cathinone-related compounds using cyclohexanone as Schiff-base derivatizing reagent was developed. The method is capable of separating the 13 cathinone compounds tested with baseline resolution in less than 11 minutes. Primary and secondary amines will react with cyclohexanone via a Schiff-base reaction to form two distinct different types of derivatives. Tertiary amines, such as diethylpropion and methylpseudoephedrine, do not react with cyclohexanone to form a derivative and also do not interfere with the method. Cyclohexanone forms an imine derivative with primary amines and enamine derivatives with secondary amines. Cathinone forms both an imine and enamine when it is derivatized using cyclohexanone. Due to the presence of the *β*-hydroxyl functional group, ephedrine and pseudoephedrine (both being secondary amines) react with cyclohexanone to form oxazolidines. The reactions are all reproducible and show good chromatographic behavior on the gas chromatographic column stationary phase tested.

GC/MS data was acquired using a quadrupole Mass-Selective Detector (MSD). The gas chromatographic oven temperature parameters were set with an initial temperature of 120 C which then increased 15 C/min to 250 C for a total run time of 10.67 minutes. The column used was a 30m x 0.25mm x 0.25µm phenylmethylsilicone capillary column (Rxi® -5Sil MS) using Helium as a carrier gas with a linear gas velocity of 38cm/sec. Methanol was used as the solvent and a sample volume of 1µL was injected in the split mode with a split ratio of 50:1. A retention time optimization study provided the most advantageous separation conditions.

Using cyclohexanone to form the Schiff-base derivative, a more complicated mass spectrum can be created and used to identify and differentiate similar cathinone compounds. The increased fragmentation also aids in providing more structural information about the compound. This method can be used to screen unknown samples and identify multiple cathinones in one sample.

Forensic Science, Forensic Chemistry, Cathinones

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