GC-MS and GC-IR Analysis of Methcathinone Analogs

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Abstract

Methcathinone is a psychoactive stimulant, structurally similar to methampthetamine. Methcathinone is a schedule 1 drug under the Controlled Substances Act. Due to the high control, many analogs are being produced to bypass the legal system. The problem faced by drug analysts in identifying these analogs is the similarity between them. GC-MS is considered the gold standard in drug identification; however its use is limited in the analysis of these analogs due to similar retention times and mass spectra. GC-IR has been shown previously to provide more accurate drug identification. This research provided examples of the problems faced when using GC-MS and how GC-IR can be used to accurately identify the analogs.

Introduction

Designer drug usage has increased in modern times due to the Controlled Substance Act.
These drugs have minor structural changes in order to produce similar pharmacological effects while bypassing DEA regulations.
Methcathinone analogs are modified in three regions: the alkyl side chain, the amino group and the aromatic ring.

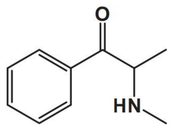

Figure 1. The structure of Methcathinone

Only three methcathinones analogs have been scheduled under the Controlled Substances Act: 4-methylmethcathinone, 3,4-methylenedioxymethcathinone and methylenedioxypyrovalerone (MDPV). GC-IR has been used to analyze numerous drugs and is a better method of identification for designer drugs since it does not require derivatization.

Structures of Methcathinone Analogs

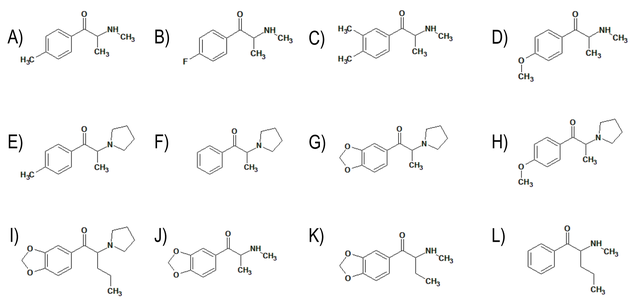

Figure 2. Structure of: A) 4-methylmethcathinone, B) 4-fluoromethcathinone, C) 3,4-dimethylmethcathinone, D) 4-methoxymethcathinone, E) 4-methyl-α-pyrrolidinopropiophenone, F) α-pyrrolidinopropiophenone, G) 3,4-methylenedioxy-α-pyrrolidinopropiophenone, H) 4-methoxy-α-pyrrolidinopropiophenone, I) Methylenedioxypyrovalerone, J) 3,4-methylenemethcathinone, K) Butylone, L) Pentedrone

Materials and Methods

All drug standards were purchased commercially from Cayman Chemical ®. The samples were made by dissolving 1 mg/mL to 3 mg/mL of the standard in chloroform.

GC-FID-IR

Instruments: Agilent 6890N GC ®
Thermo Scientific Nicolet 6700 FTIR ®
Thermo Scientific GC/IR Interface ®
GC Oven: 115°C, 20.0°C/min, 290°C (4.00 min), 50°C
Injection Port: 225°C Splitless Mode
Carrier Gas: Helium (2.0 mL/min)
Column: HP-1 Methyl Siloxane Capillary (30.0m x 320 µm x 1.00 µm)
IR Number of Scans: 16 IR Range: 4000-650 cm^{-1}
IR Resolution: 8-16 cm^{-1} IR Aperture: 150
Detector Flow Cell: 280°C Transfer Line: 280°C

GC-MS

Instruments: Agilent 7890A GC ® coupled with an Agilent 5975C MSD ®
Mass range analyzed: 40-500 m/z
Inlet: 250°C
Oven: 70°C (2.00 min), 20.0°C/min, 270°C

Results and Discussion

Table 1. The characteristic IR bands found in the methcathinone analogs.

Compound	Characteristic IR Bands (S=strong, M=medium, W=weak)																
3,4-dimethylmethcathinone	3025 (W)	2978 (S)	2936 (S)	2884 (M)	2805 (W)	1694 (S)	1606 (M)	1568 (W)	1460 (W)	1299 (W)	1240 (M)	1160 (M)	1124 (M)	976 (M)	829 (W)	767 (W)	
Butylone	2971 (W)	2942 (W)	2887 (W)	2806 (W)	1690 (M)	1613 (W)	1486 (M)	1437 (M)	1347 (W)	1246 (S)	1097 (W)	1049 (M)	943 (W)	805 (W)			
Pentedrone	3070 (W)	3036 (W)	2968 (S)	2941 (S)	2883 (M)	2806 (W)	1696 (S)	1596 (W)	1448 (W)	1242 (W)	1208 (M)	1179 (W)	1131 (W)	988 (W)	770 (W)	697 (M)	
2-methoxymethcathinone	3074 (W)	2945 (M)	2803 (W)	1699 (S)	1595 (M)	1480 (S)	1443 (M)	1279 (M)	1242 (S)	1185 (M)	1119 (M)	1026 (M)	961 (M)	753 (M)			
3-methoxymethcathinone	3077 (W)	2977 (M)	2948 (M)	2808 (W)	1698 (M)	1588 (M)	1480 (M)	1429 (M)	1258 (S)	1166 (W)	1048 (M)	992 (W)	764 (W)				
4-methoxymethcathinone	3081 (W)	2975 (M)	2946 (M)	2851 (W)	2811 (W)	1691 (S)	1600 (S)	1507 (M)	1464 (W)	1416 (W)	1296 (M)	1253 (S)	1169 (S)	1037 (M)	960 (M)	840 (W)	766 (W)	701 (W)
2-methylmethcathinone	3067 (M)	3024 (M)	2977 (S)	2940 (S)	2806 (W)	1700 (S)	1597 (W)	1459 (M)	1378 (W)	1290 (W)	1218 (M)	1191 (M)	1128 (M)	957 (M)	733 (M)		
3-methylmethcathinone	3058 (W)	2978 (M)	2934 (M)	2807 (W)	1697 (S)	1590 (W)	1480 (W)	1441 (W)	1373 (W)	1249 (M)	1159 (M)	1010 (W)	972 (W)	763 (M)			
4-methylmethcathinone	3032 (W)	2979 (M)	2935 (M)	2887 (M)	2803 (W)	1694 (S)	1607 (M)	1475 (W)	1371 (W)	1178 (M)	1129 (M)	958 (M)	824 (W)	764 (M)	702 (W)		
2-fluoromethcathinone	N/A																
3-fluoromethcathinone	3070 (W)	2979 (M)	2942 (W)	2807 (W)	1702 (S)	1586 (M)	1481 (W)	1439 (M)	1255 (S)	1147 (W)	981 (W)	841 (W)	766 (W)				
4-fluoromethcathinone	3071 (W)	2980 (M)	2940 (W)	2809 (W)	1697 (S)	1598 (S)	1504 (M)	1235 (S)	1156 (M)	961 (M)	844 (M)	765 (W)	698 (W)				
	cm^{-1}																

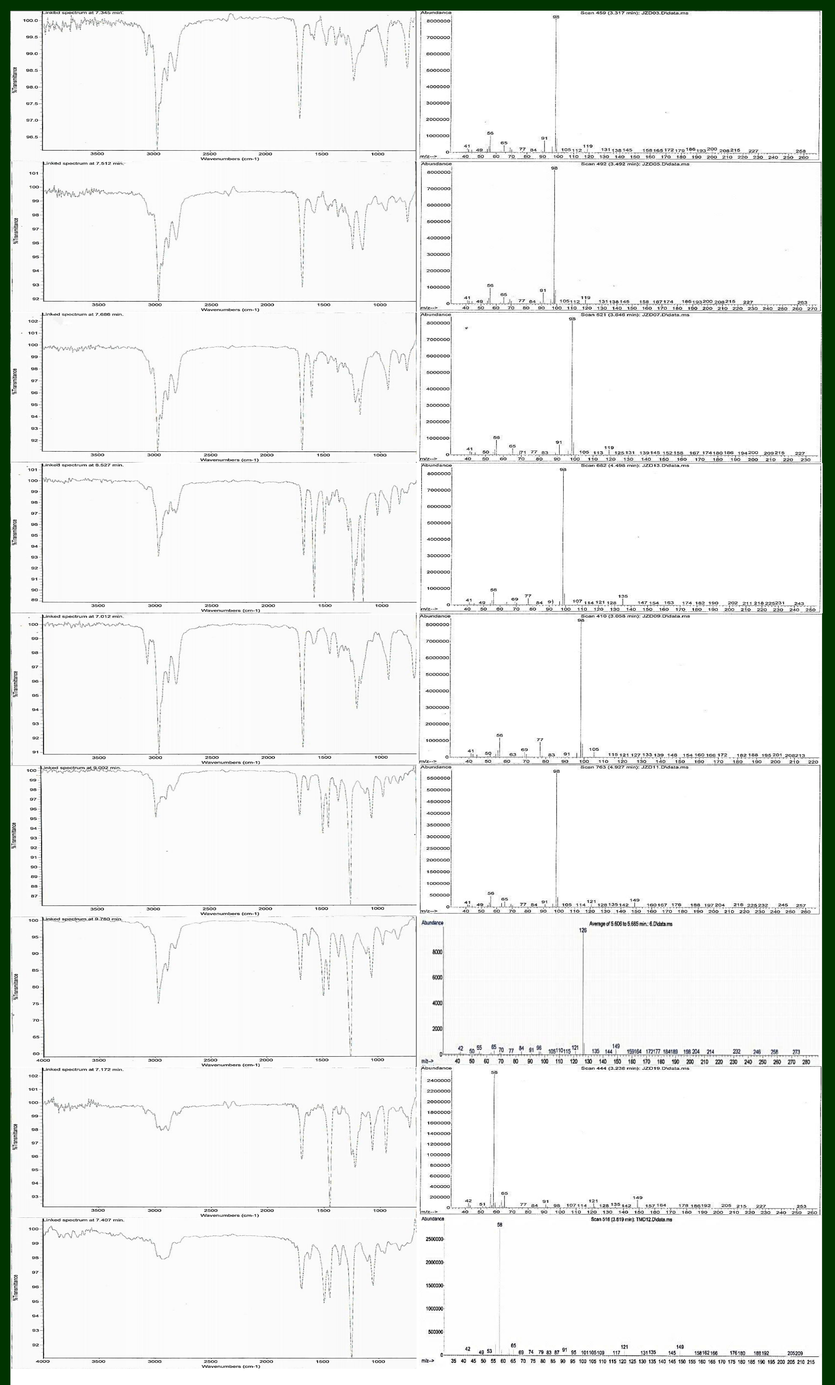

Figure 3. GC-IR Spectrum and GC-MS Spectrum for: 2-methyl-α-PPP, 3-methyl-α-PPP, 4-methyl-α-PPP, 4-methoxy-α-PPP, α-PPP, 3,4-methylenedixoy-α-PPP, MDPV, 2,3-methylenedioxymethcathinone, 3,4-methylenedioxymethcathinone.

Table 2. Mass spectra peaks of interest for the methcathinone analogs.

Compound	MS Peaks of Interest (* represents the base peak)							
3,4-dimethylmethcathinone	42	58*	77	105	133			
Butylone	42	57	65	72*	91	121	149	192
Pentedrone	44	51	57	70	77	86*	105	148
2-methoxymethcathinone	42	51	58*	77	92	121	135	
3-methoxymethcathinone	42	51	58*	64	77	92	107	135
4-methoxymethcathinone	42	50	58*	77	92	107	135	
2-methylmethcathinone	42	51	58*	65	91	119		
3-methylmethcathinone	42	50	58*	65	91	119		
4-methylmethcathinone	42	51	58*	65	91	119		
2-fluoromethcathinone	42	50	58*	75	95	123	161	
3-fluoromethcathinone	42	50	58*	75	95	123	166	
4-fluoromethcathinone	42	50	58*	75	95	123	166	
	(m/z)							

Conclusion

All of the methcathinone analogs featured bands between 2967-2980, 2803-2818, and 1689-1702 cm^{-1}.
The analogs that contained a methylenedioxy group on the aromatic ring contained bands between 1344-1355, 1245-1253, 1049-1067, and 944-946 cm^{-1}.
The analogs containing the pyrrolidine ring at the amino group contained bands between 927-945 cm^{-1}.
The fluoromethcathinones contained bands between 1147-1156 cm^{-1}.
The analogs containing the methoxy group on the aromatic ring featured bands in the range 1242-1258 cm^{-1}.
The GC-IR spectrum obtained for the methcathinone analogs provided a more unique identification than GC-MS in every set of compounds except for MDPV and 3,4-methylenedioxy-α-PPP.
The range from 690-1700 cm^{-1} provided valuable information when analyzing positional isomers.
The 2-positional isomers contained bands between 733-753 and 957-961 cm^{-1}.
The 3-positional isomers contained bands in the ranges 753-766 and 972-992 cm^{-1}.
The 4-positional isomers contained bands in the ranges 760-775, 958-961, and 1600-1607 cm^{-1}.
GC-IR provides a better identification of methcathinone analogs than GC-MS.

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Acknowledgements

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