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RESEARCH ARTICLE

Unusual Detection of Monosodium Glutamate as an Adulterant in Mephedrone Sample: Confirmation by Raman Spectroscopy and Composition by Simple Solubility Differences

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Abstract: Mephedronehydrochloride (4-methylmethcathinone. HCl / 2-(methylamino)-1-(*p*-tolyl) propan-1-one hydrochloride) adulterated with monosodium glutamate (Ajinomoto) were detected and confirmed by very simple and accurate Raman spectroscopy. Further adulteration ratio was determined by solubility differences of mephedrone and ajinomoto in methanol. The recovery of each component were obtained efficiently, thus this study provides simple and effective method of recognition and judgment of composition of mephedrone and ajinomoto.

Keywords: Mephedrone, Ajinomoto, Raman spectroscopy, Monosodium glutamate, Solubility

Introduction

Recently mephedrone has been emerged as alternatives for the most abused stimulants such as amphetamine, methamphetamine (MA), methylenedioxymethamphetamine (MDMA)[1,2] *etc*. and has been banned and regulated under control substances act in many countries across the world[3]. In India from February 2015 mephedrone has been included in the Narcotic and Psychotropic substances act due to several hundred illegal mephedrone seizers registered by the police agencies, its addiction and adverse effects. Mephedrone is a white substance and sold most commonly as crystals or powder as hydrochloride salt. Few reports comment the consignments of the many abused drugs are either as adulterated or completely different from the mentioned compound with dangerous chemical compounds of similar appearances[4]. In April 2015 we unusually detected monosodium glutamate as an adulterant in mephedrone sample. Ajinomoto is a well-known non-essential amino acid utilized as a flavour enhancement agent in food stuffs. Due to the similarities in appearance of crystals and water solubility of monosodium glutamate and mephedrone it was superficially difficult to detect adulteration of ajinomoto in mephedrone by the consumers and even police agencies. Consequently many

police enforcement agencies were seized consignment of ajinomoto as mephedrone[5]. It was then necessary to develop easy protocol to detect, confirm and differentiate the same for routine analysis. Raman spectroscopy is a non-destructive technique which provides a "fingerprint" spectrum of chemical compounds and very accurate and powerful technique in the forensic analysis.

Experimental

We scanned the reference ajinomoto, mephedrone and uniformly grinded portion of sample under examination on Renishaw Raman spectrometer and recorded spectral graph with specific operating parameters (Table 1, Figure 1 to 3). The observations of specific peaks are recorded in Table 2.

Table 1. Raman operating conditions

Make of Raman spectrometer	Renishaw, Invia Raman Microscope
Laser source used	785 nm edge
Laser power used	5 %
Lens for imaging	20x
Beam path	Grating
Acquisition time and accumulations	10 and 1

Table 2. Some specific Raman shifts of sample, Ajinomoto and Mephedrone

S. No.	Raman shifts/cm^{-1} of Mephedrone	Raman shifts/cm^{-1} of Ajinomoto	Raman shifts/cm^{-1} of sample	
			Mephedrone	Ajinomoto
1	250	174		173
2	292	354		
3	365	525		526
4	470	601		601
5	522	660		
6	635	790	634	
7	732	855		855
8	801	874	801	873
9	897	940		940
10	976	1000		1001
11	1004	1053	1001	
12	1045	1093		
13	1164	1157		1160
14	1185	1279		
15	1211	1315		1314
16	1245	1338		1338
17	1380	1398	1602	1399
18	1603	1431	1683	1431
19	1684			

After detection and confirmation of ajinomoto in the sample, it was necessary to determine the composition of the adulterant in the sample and for that purpose very simple solubility behaviour of the both compounds were utilised. Taking in to consideration the fact that ajinomoto is insoluble in methanol whereas mephedrone is soluble in methanol, the logical way to separate both components was by mixing with methanol, thus following procedure was adopted. The recovery studies were done on known compositions of ajinomoto and mephedrone mixtures by w/w method and then on the sample and summarised in Table 3.

Table 3. Recovery observations

S. No.	Weight of mephedrone in mgs	Weight of ajinomoto in mgs	Weight obtained after recovery in mgs		% Compositions obtained		*Actual % Composition of M : A
			Mephedrone	Ajinomoto	Mephedrone	Ajinomoto	
1	100	100	105	95	53	47	~50:50
2	150	100	157	93	62.8	37.2	~60:40
3	250	150	264	136	66	34	~ 62:38
4	100	200	106	194	35.3	64.6	~ 33:67
	Sample in question(300)		140	160	46.6	53.3	~ 44:56

**Corrected composition of mephedrone: Ajinomoto by considering 3% incorporation factor of ajinomoto*

Representative procedure for the recovery study

100 Milligrams of ajinomoto and 150 milligrams of mephedrone was mixed with each other, mixture were taken in a previously weighed glass beaker, few mL of methanol was added and shaken well to dissolve the mephedrone completely, after settlement of crystals filter supernatant liquid through Whatmann filter paper no.1 in preweighed Petri dish. The same procedure was repeated for another three times and methanol were evaporated over two consecutive nights at r.t. then both beakers and petridish were further dried in oven for one and half hour at 70 ^{0}C. Then final weights were recorded. After substracting initial weights from final weights the recovery of mephedrone by this method were found to be 157 milligrams and that of ajinomoto were 93 milligrams.

Results and Discussion

Unique fingerprint peaks of Raman spectra in the region of 100-1900 cm^{-1} were explored for the identification and confirmation of reference ajinomoto and mephedrone which clearly reflect their characteristics peaks in the spectrum and can be used for differentiation. The observations obtained from the spectrum suggest that though there is combination of these two compounds; each compound reflects their characteristic peak in the Raman spectrum and thus showing their remarkable existence (Figure 1 to 3 and Table 2).

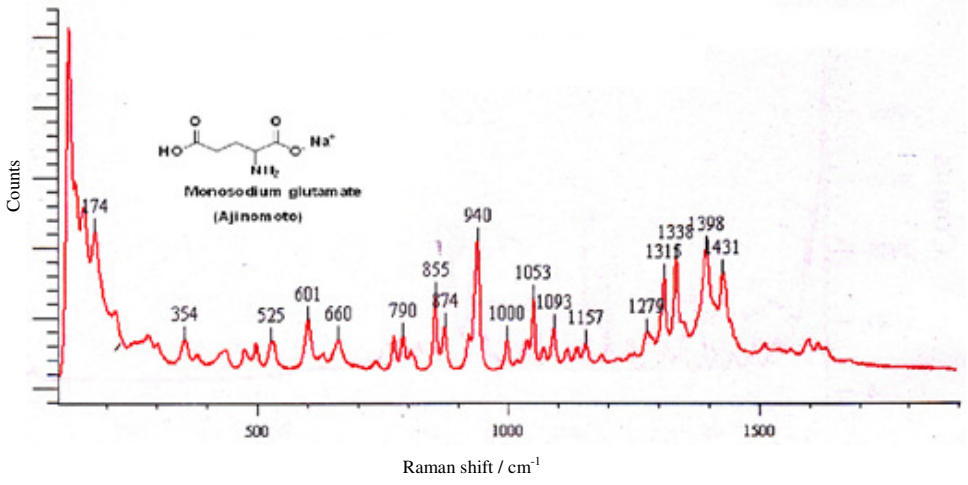

Figure 1. Raman spectrum of referene monosodium glutamate

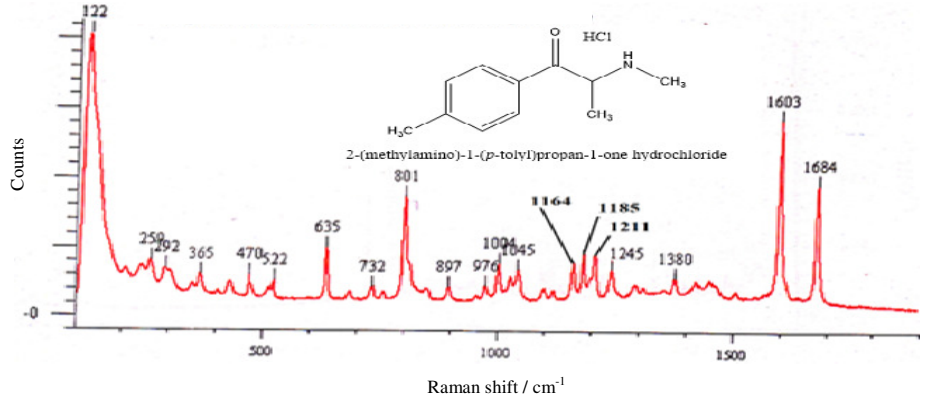

Figure 2. Raman spectrum of referene mephedrone

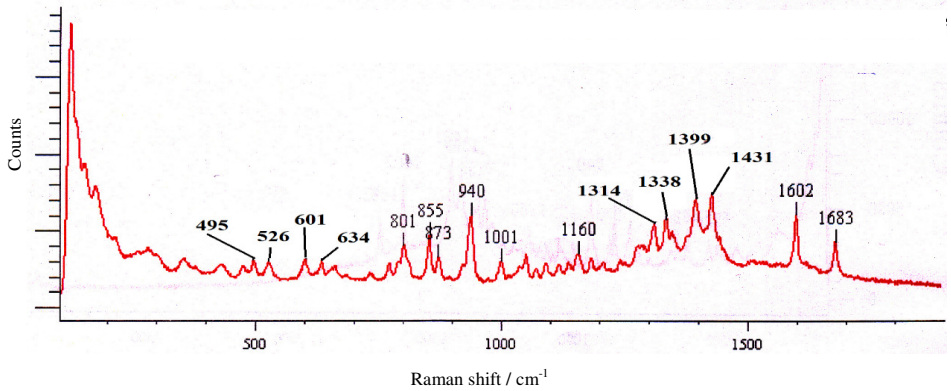

Figure 3. Raman spectrum of sample in question shows peak of both ajinomoto and mephedrone

The recoveries of both compounds were expected but it was observed that during transferring the dissolved mephedrone solution in other gadget there is incorporation of ajinomoto in the methanol solution. This observation were reflected by thin layer chromatography and thus we utilised weighing observation made in the recovery study and conclude that ~3% of ajinomoto was incorporated in each case of recovery, this fact can treated as an incorporation factor of ajinomoto and can be corrected when finding the actual composition (Table 3). Further it was observed that the recovered samples shows same fingerprint Raman spectra for ajinomoto and mephedrone (In case of recovered mephedrone no interference of ~3% ajinomoto were detected)

Conclusion

Simple non-destructive and reliable method of detection and confirmation for ajinomoto in mephedrone samples was discussed. The protocol provides an access to evaluate the composition of mixtures made by ajinomoto and mephedrone.

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