



Separation of Methamphetamine from Dimethyl Sulfoxide by Solubility Differences and Identification, Confirmation by Raman Spectroscopy

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Abstract

Narcotic drug with addition of neutral substances is common practice in India. Paracetamol, phenacetin and lignocaine are commonly used for addition in narcotic drugs like heroin and cocaine. Dimethyl sulfoxide is a neutral substance used in methamphetamine hydrochloride. Methamphetamine is a stimulant popular in the illicit drug market known as ice drug. The pure crystalline Methamphetamine and Dimethyl sulfoxide easily detected and identified by Raman spectroscopy. However, mixed Raman spectra was obtained from powdered sample. The identification of methamphetamine in Raman Spectra, was done by dissolving methamphetamine hydrochloride dissolving in methanol and precipitated in ether, dimethyl sulfoxide remains in methanol -ether mixture. Raman spectra of precipitated drugs confirm the methamphetamine. Solubility differences in the two solvents made separation easy and Raman spectroscopy confirms the presence of methamphetamine hydrochloride and dimethyl sulfoxide.

Keywords: Neutral substances; Narcotic Drugs; Adulterant; Methamphetamine; Dimethyl sulfoxide; Solubility; Raman Spectra

Introduction

All opiates have a long history of mixing neutral substances by drug traffickers. In India, Mumbai, the forensic analysis of heroin samples seized most of the samples mixed with paracetamol and caffeine [1].

Paracetamol is a pain relief drug easily available without a doctor's prescription in any pharmacy. In a few cases, it is found that, Alprazolam a depressant drug added in heroin sample which falls under NDPS act 1985. Despite strict legal provisions, the illegal business of narcotic drugs has been growing day by day and in recent 5 years the increase of

narcotic cases has widely increased. But with the increase in the number of cases, the increase in adulterated drugs is also seen in a lot of street samples. In 2015, Mephedrone, a simulant drug added in NDPS Act 1985 is widely seen in narcotic seizures, as soon as its seizure increases a pure sample was adulterated with Aijnomoto a Chinese salt whose appearance is quite similar to Mephedrone [2]. Phenacetin is another popular adulterant in Cocaine sample [3]. On the other hand, similar to this scenario, Methamphetamine another popular stimulant with a famous street name 'ice drug' is adulterated with Dimethyl Sulfoxide, a dietary supplement and it is a common cutting agent for ice drug as its appearance is similar to Methamphetamine hydrochloride. Due to addition

of neutral substances identification of samples is not an easy task and forensic experts have to perform a different test to rule out the possibility of said drug. However, the identification of such adulterant, cutting agents and place of seizure, accused or organization involved in crime, such detail profiling of crime can help to establish evidence to track down the criminal organization or traffickers involved in synthetic drug preparation [4]. Detailed analysis of street samples can throw light on a new designer drug that is not controlled in the drug market and is used as a narcotic drug of abuse by a drug cartel to escape from the law, such new psychoactive substances can be included in controlled substances by further study and its illegal use as a drug of abuse can be controlled by legal provisions [5].

Raman Spectroscopy is an easy and reliable technique for different types of drugs. It is a non-destructive technique and for a pure sample there is no special pre-treatment is required hence sample is unchanged [6,7], and substituted cathinone has been also easily differentiated by Raman spectroscopy [8]. Raman spectroscopy can be successfully employed for narcotic drug identification, even this technique gives the idea of an adulterated sample.

Methodology

Dimethyl sulfone and Methamphetamine hydrochloride both are easily soluble in methanol however in ether its vice versa, dimethyl sulfone is soluble but Methamphetamine hydrochloride is practically insoluble. This solubility similarity and difference in different solvents made their separation from mixture easy. Methamphetamine hydrochloride can be precipitated in ether from methanol.

Model of Raman Spectrophotometer	Renishaw, Invia Raman Microscope
Laser Source	785 nm Edge
Laser Power	10%
Lens Power	20X
Beam Path	Grating
Acquisitions:	10
Accumulations	1

Table 1: Values of Model of Raman Spectrophotometer and Renishaw, Invia Raman Microscope in Different Paths.

Examination Parameters

A sample previously analysed by Mass spectroscopy shows the presence of Dimethyl sulfone and methamphetamine was taken and scanned. Raman shifts are recorded. From this mixed sample after homogenous mixing, weighed accurately 500 mg of sample from the whole sample and transfer to a clean test tube of about 25ml capacity then 4ml Methanol was added to it and shaken thoroughly to dissolve completely. Then 16ml of ether was added to this test tube and white precipitation took place in the test tube. This precipitate is filtered through whatman filter paper no.1, and the residue remained on the filter paper is kept overnight at room temperature for drying. On the second day dried sample was taken and scanned on a Raman spectrophotometer. Along with this, we scanned the reference sample of Dimethyl sulfone, and Methamphetamine Hydrochloride as well. All the Raman frequencies (Raman shift/cm-1) of respective samples are recorded in (Table 2).

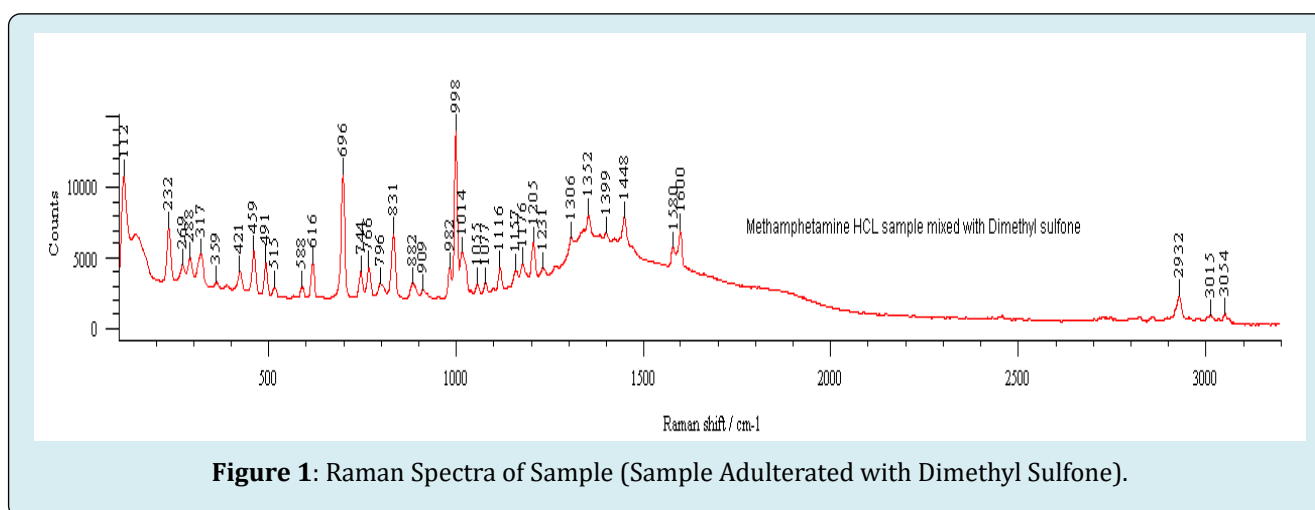


Figure 1: Raman Spectra of Sample (Sample Adulterated with Dimethyl Sulfone).

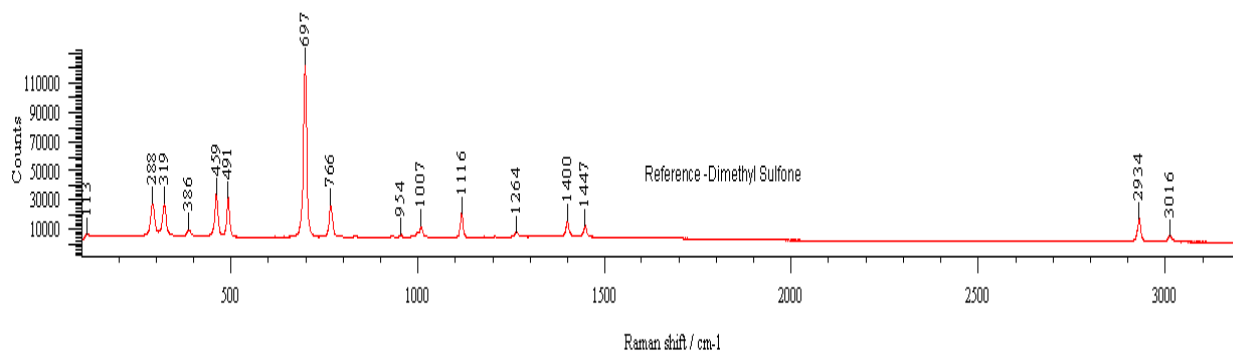


Figure 2: Raman Spectra of Dimethyl Sulfone.

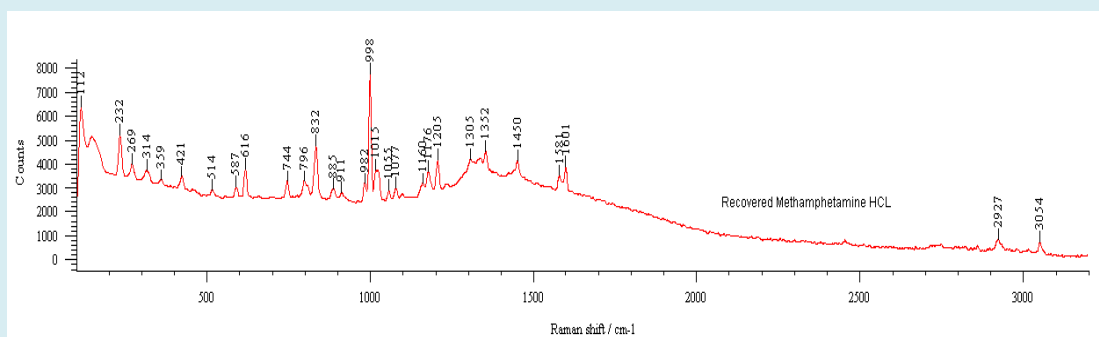


Figure 3: Raman Spectra of Recovered Methamphetamine Hydrochloride from Sample After Precipitated in Ether.

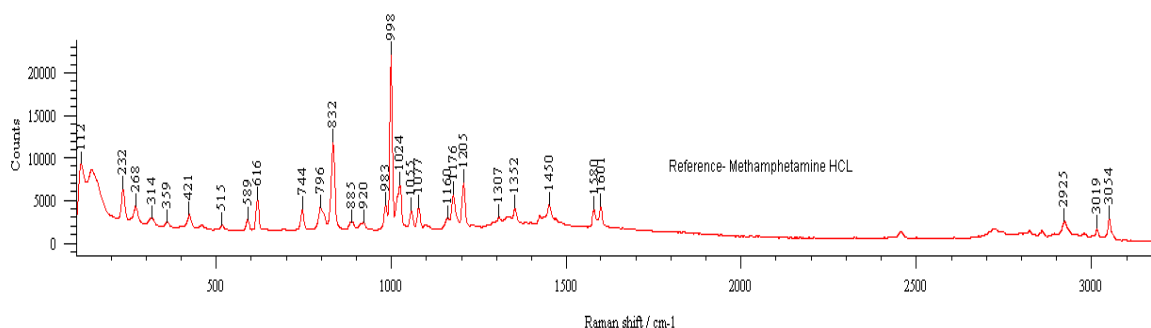


Figure 4: Raman Spectra of Methamphetamine Hydrochloride.

Results and Discussion

Mixed sample shows Raman frequencies of both compound, Methamphetamine hydrochloride and Dimethyl sulfone (Figure 1). when this spectrum compared with Raman spectra of Methamphetamine hydrochloride, characteristic Raman frequency 616nm, 832nm, 998nm, 1205nm, 1580nm, 1601 nm (Figure 4) in the sample, it

gives the idea that sample might contains Methamphetamine and other adulterant. While other Raman frequencies with remarkable presence in the sample are 459nm, 491nm, 696nm, 766nm, 1116nm and 1448 nm which corresponds to dimethyl sulfone (Figures 1 & 3). After precipitation in ether; the separated and dried sample gives spectra which shows absence of Raman frequencies corresponds to Dimethyl sulfone and the spectra is matched with reference

spectra of Methamphetamine this confirms presence of Methamphetamine hydrochloride (Figures 3 & 4). All results are tabulated in (Table 2).

Sr .no.	Raman shift/cm-1 of Mixed Sample	Raman shift/cm-1 of reference Dimethyl Sulfone	Raman shift/cm-1 of Recovered Methamphetamine Hydrochloride	Raman shift/cm-1 of reference Methamphetamine Hydrochloride
1	232	-	232	232
2	269	-	269	268
3	288	288	-	-
4	317	319	314	314
5	359	386	359	359
6	421	-	421	421
7	459	459	-	-
8	491	491	-	-
9	515	-	514	515
10	588	-	587	589
11	616	-	616	616
12	697	697	-	-
13	744	-	744	744
14	766	766	-	-
15	796	-	796	796
16	831	-	832	832
17	882	954	885	885
18	909	-	911	920
19	982	-	982	983
20	998	-	998	998
21	1014	1007	1015	1024
22	1055	-	1055	1055
23	1077	-	1077	1077
24	1116	1116	-	-
25	1157	-	1160	1160
26	1176	-	1176	1176
27	1205	-	1205	1205
28	1231	1264	-	-
29	1306	-	1305	1307
30	1352	-	1352	1352
31	1399	1400	-	-
32	1448	1447	1450	1450
33	1580	-	1581	1580
34	1600	-	1601	1601
35	2932	2934	2927	2925
36	3015	3016	-	3019
37	3054	-	3054	3054

Table 2: Raman Shift/Cm-1 of Mixed Sample, Dimethyl Sulfone, Methamphetamine Hydrochloride and Methamphetamine Hydrochloride.

Conclusion

Raman spectroscopy is an accurate technique for identification of narcotic drugs, even if sample is adulterated with other substances and if it is Raman active, a powdered sample give the idea of mixture and possible narcotic drugs in the sample. It can be successfully employed for such street samples which are adulterated with different either neutral substances or hazardous pharmaceutical preparation like paracetamol, Lidocaine. Raman spectroscopy is valuable technique for confirmation and identification of narcotic drugs even in mixture and if a sample is separated from mixture, a very small sample quantity is required for Raman spectroscopy to the confirmation of a narcotic drug in adulterated sample.

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