Identification of Delorazepam in Suspected Methaqualone Contraband: A Case Study

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Abstract

The present case study reports the analysis of seized narcotic drug received from investigation agency in Mumbai. The drug was suspected to be methaqualone which falls under NDPS act 1985. Analysis of the drug was carried out using HPTLC, GC-MS, UV and Raman spectroscopy techniques. These complimentary techniques revealed and confirmed the presence of delorazepam in the suspected sample while methaqualone was found to be absent. The case study highlights the importance of analytical techniques in identifying the narcotic drug in the seized sample.

Keywords- Delorazepam, Methaqualone, NDPS act 1985, Forensic sample, Commercial Quantity

1.Introduction

Analytical techniques in forensic sciences play a crucial role in the identification and characterization of drugs, which is essential for law enforcement and legal proceedings. These techniques, such as Gas Chromatography-Mass Spectrometry (GC-MS), High-Performance Thin Layer Chromatography (HPTLC), and Fourier Transform Infrared Spectroscopy (FTIR) allow forensic analysts to accurately detect and quantify the presence of controlled substances in various samples. By providing detailed chemical fingerprints of substances, these methods enable the distinction between closely related compounds and the precise determination of drug purity. This level of specificity is critical not only for identifying the type of drug present in a sample but also for tracing its origin and understanding its potential effects, thereby aiding in criminal investigations, and contributing to the establishment of forensic evidence in court

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cases. The present paper demonstrates the role of analytical techniques in identifying the suspected drug received from the investigation agency.

Among the various analytical techniques, GC-MS is crucial in forensic science for several reasons. It provides highly accurate and reliable results, which are essential in legal contexts. It can identify and quantify various substances, including drugs, toxins, and explosives, with great precision. This accuracy helps in determining the cause of death, identifying unknown substances, and linking evidence to crime scenes or suspects. GC-MS results are widely accepted in courts due to their robustness and reliability. The technique's credibility and the ability to withstand legal scrutiny make it indispensable in forensic investigations. Maurer et al.[1] have discussed the identification and differentiation of benzodiazepine drugs in urine samples using GC-MS. Maudens et al.[2] have reported unexpected benzodiazepine findings in three forensic toxicological cases using GC-MS technique. Other case studies, such as the detection of dextropropoxyphene in a suspected opium sample and the detection of alprazolam in a suspected heroin sample, have been documented by our laboratory.[3, 4]

Other analytical techniques such IR, Raman, and UV also play an important role in forensic investigations: Neville et al.[5] have described the vibrational spectra (IR and Raman) of benzodiazepines, including Raman spectra wavenumbers for some of these compounds. Additionally, Bumbrah et al.[6] provided an overview of the developments in the analysis of drugs of abuse using Raman spectroscopy for forensic purposes and the application of this instrument in characterizing drugs. Atole et al.[7] detailed a UV spectrophotometric method for drug analysis, including theoretical and mathematical expressions and some applications.

The Forensic Science Laboratory (FSL) receives samples seized under the Narcotic Drugs and Psychotropic Substances (NDPS) Act 1985. These narcotic drugs or psychotropic substances are typically confiscated by investigating officers based on tips. Initial identification of these samples is performed on-site using color tests with available kits. These preliminary tests are not confirmatory; hence, the samples are sent to the FSL for further analysis. The investigation agency in Mumbai seized 485 g drugs suspected as methaqualone which falls under NDPS act 1985. Our laboratory had received 4.83 g of this suspected sample of methaqualone for further examination.

The United Nations Office of Drugs and Crime (UNODC) has recommended various methods for analyzing the narcotic drugs[8,9]. Systematic analysis of suspected drug using various analytical techniques in our laboratory revealed that suspected sample was not methaqualone

but Delorazepam which also falls under NDPS act 1985. The commercial quantity was decided on the basis of Potency of the drugs i.e. the commercial quantity is less for more potent drug.

2 Experimental

2.1 Chemicals and reagents

AR grade Benzene, acetonitrile, methanol, potassium iodide, bismuth sub nitrate and acetic acid of Sigma Aldrich were used for the analysis of the sample. Draggendorff's reagent was prepared by using potassium iodide, bismuth sub nitrate and acetic acid which was used as spraying reagent for HPTLC plate of silica.

2.2 Procedure

The suspected methaqualone sample received was white powder. Solubility studies on it revealed that it is completely soluble in methanol and insoluble in water. Very small quantity of sample was taken on spot tile and three drops of cobalt thiocyanate reagent was added which showed no colour change indicating absence of methaqualone in sample contraband. In order to further identify and confirm the suspected drug, analysis of the sample was done using various analytical techniques. 1 mg/10mL of sample in methanol was taken for GC-MS and HPTLC analysis.

2.3 High Performance Thin Layer Chromatography

1 mg/ 10mL of standard methaqualone was taken for analysis. Standard and sample were spotted on precoated HPTLC silica plate and the plate was developed in solvent system Benzene: Acetonitrile: Methanol (8:1: 1). Thereafter, Draggendorff's reagent was sprayed on HPTLC plate.

2.4 Ultra -Violet spectrophotometer

UV spectrophotometer of Thermofisher was used. A UV spectrum of sample in methanol showed λmax at 227.9 nm and 319.7nm.

2.5 Gas Chromatography – Mass spectroscopy

GC-MS of Agillent technologies was used for analysis. 1mg/mL of sample in methanol was taken for analysis, 2 microliter sample was injected in GC and following conditions were kept for GC- MS.

Inlet temperature- 150 ^{0}C, MS temperature-230 ^{0}C, QP temperature-250 ^{0}C, Helium was used as carrier gas at 1mL/min flow, HP-5ms capillary column of 30m length. The mass spectra showed presence of delorazepam.

2.6 Raman spectra

Raman spectrometer with microscope of Renishaw technologies was used. The FT- Raman spectra of sample were recorded after the laser beam is focused on the sample operating at 783 nm.

3.0 Results and Discussion

Suspected narcotic drug methaqualone sample was analyzed using different analytical techniques. However, initial screening test showed absence of methaqualone. Further it was confirmed by HPTLC analysis which revealed absence of this drug in suspected sample (Fig. 1). 1mg/mL sample was prepared and spotted on high performance thin layer chromatography (HPTLC). The plate was developed in solvent system benzene: acetonitrile: methanol (8:1:1) and further observed under UV at 250 nm. The observations noted in Table 1 revealed that sample does not contain methaqualone. 2 µl of above sample was then injected to GC-MS, which showed sharp peak at RT- 23.62 min and the mass spectra of same peak showed mass spectrum primary ions at m/z 304, 275, 269,241,205,186,177,138,120,102,89,75,63 which exactly match with delorazepam as depicted in Figs. 2 and 3.

UV Visible spectra of sample solution in methanol showed λmax at 227.9 nm and 319.7 nm (Fig.4) which are characteristic of delorazepam it tallies with literature value 227 and 319. The sample was further analyzed using Raman spectroscopy. The spectra obtained are shown in Fig. 5. and the Raman shift data are presented in Table 2. An examination of different peaks obtained reveals presence of delorazepam in the suspected sample.

The commercial quantity and small quantity of drugs are mentioned in NDPS act 1985. Depending upon the quantity, the granting bail and/or punishment is decided by Judiciary. Commercial quantity of methaqualone is mentioned as 500 g and that of delorazepam as 100 g. The seized quantity by investigating agency was 485 g, hence the seized quantity can be considered as commercial quantity. The commercial quantity was decided on the basis of Potency of the drugs i.e. the commercial quantity is less for more potent drug. The seized quantity of delorazepam was more potent than that of methaqualone.

Conclusion

The suspected methaqualone drug received from the investigating agency was analyzed in our laboratory using HPTLC, GC-MS, UV, and Raman spectroscopy techniques. The results

revealed the presence of Delorazepam and not methaqualone, in the suspected sample. This work demonstrates the crucial role of analytical techniques in forensic cases.

Figures:

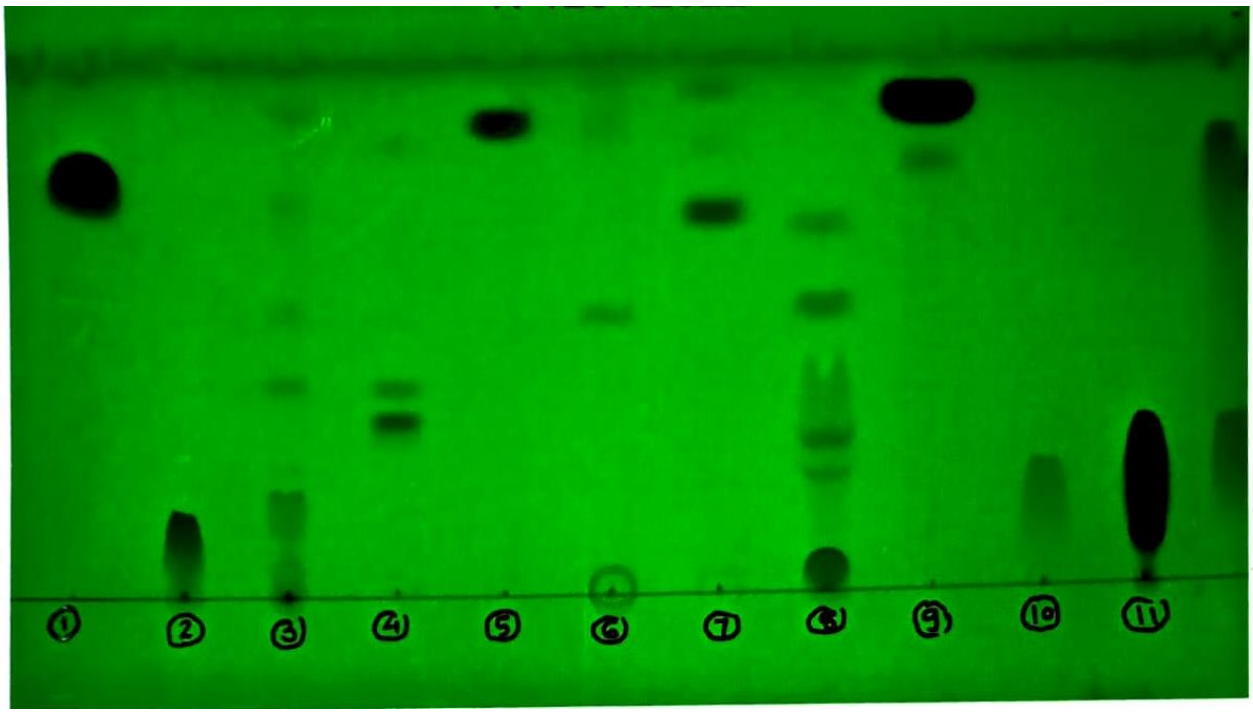


Fig. 1: HPTLC plate of contraband sample

1- **Contraband sample, 2-Control Morphine, 3-Opium, 4-Control Alprazolam,5-Control Diazepam, 6-Control Lorazepam, 7-Control Nitrazepam, 8-Control Cocaine, 9-Control Methaqualone, 10-Control Methamphetamine, 11-Control Ketamine**

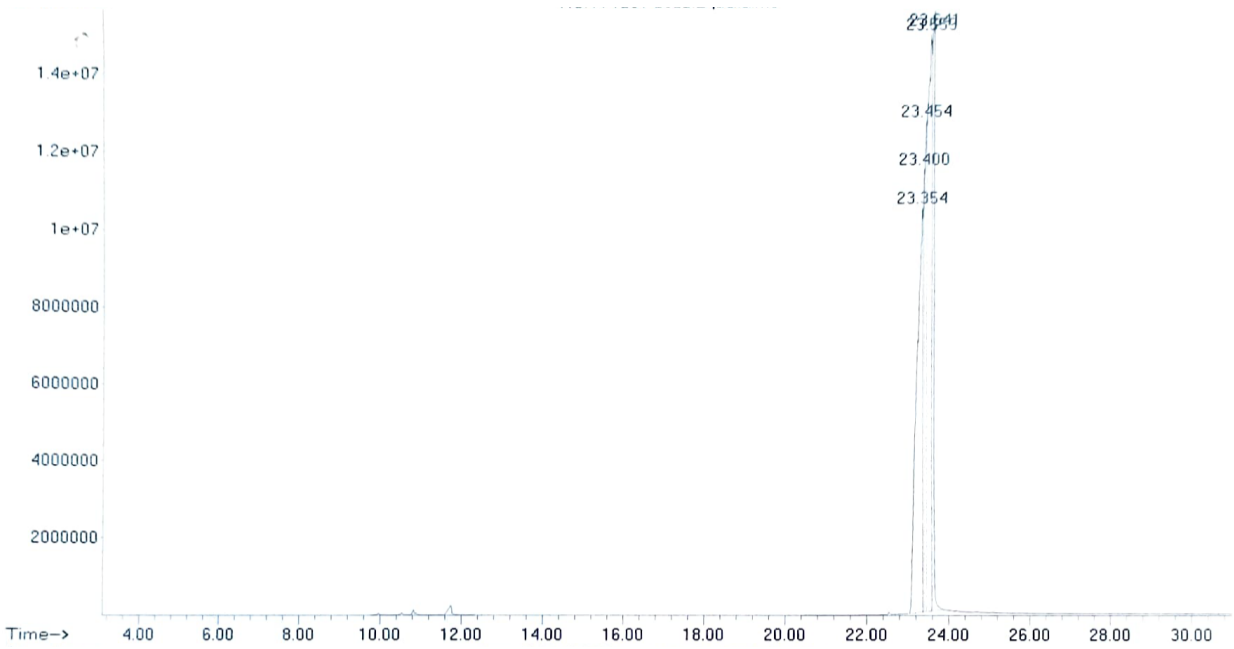


Fig. 2 : Gas Chromatogram of contraband sample

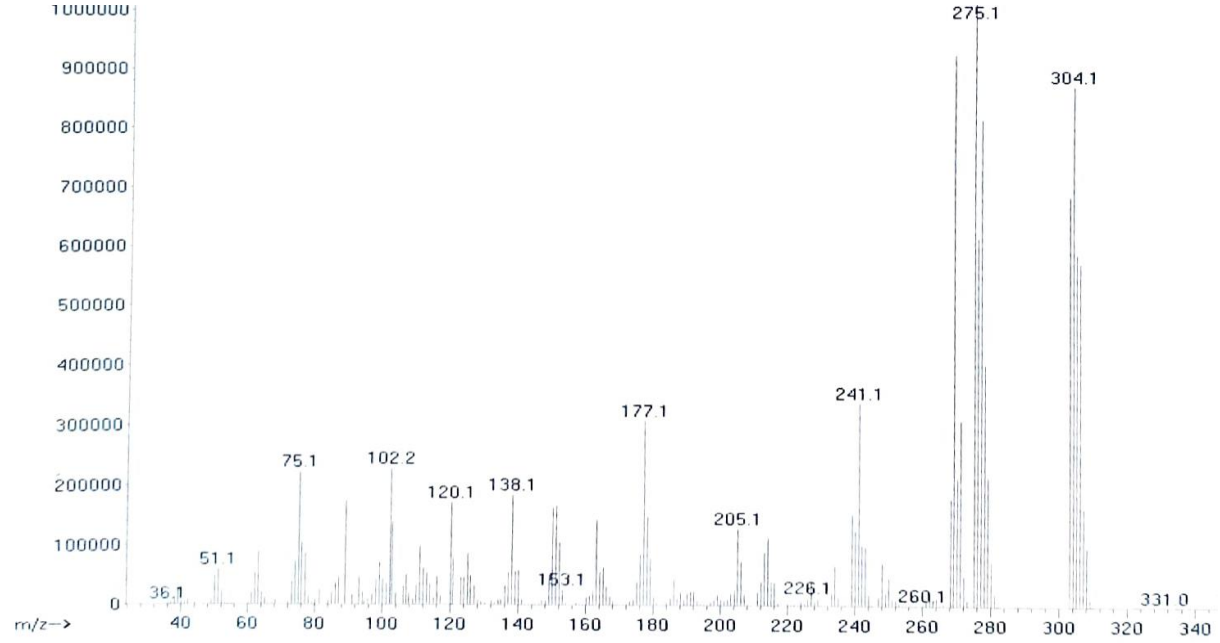


Fig. 3 : Mass spectra of contraband sample

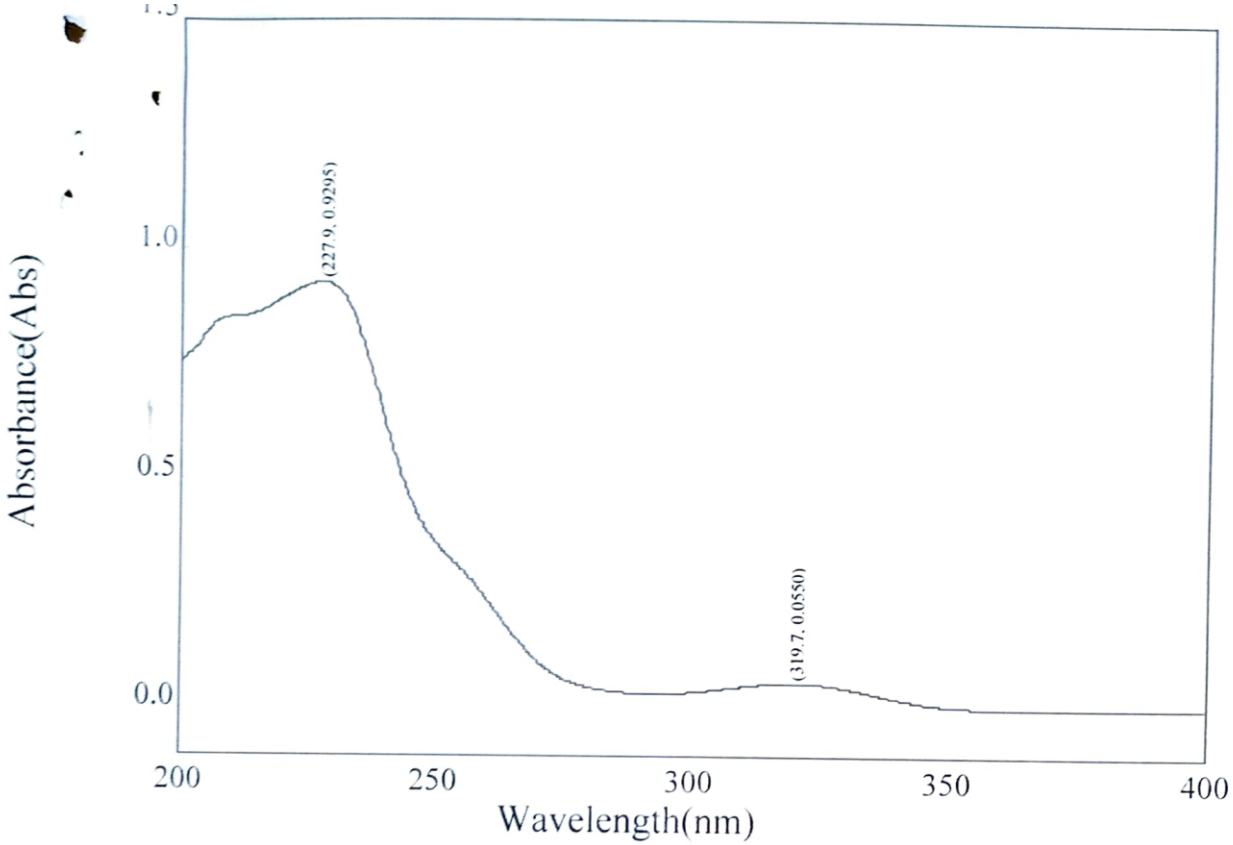


Fig. 4 : UV spectra of contraband sample

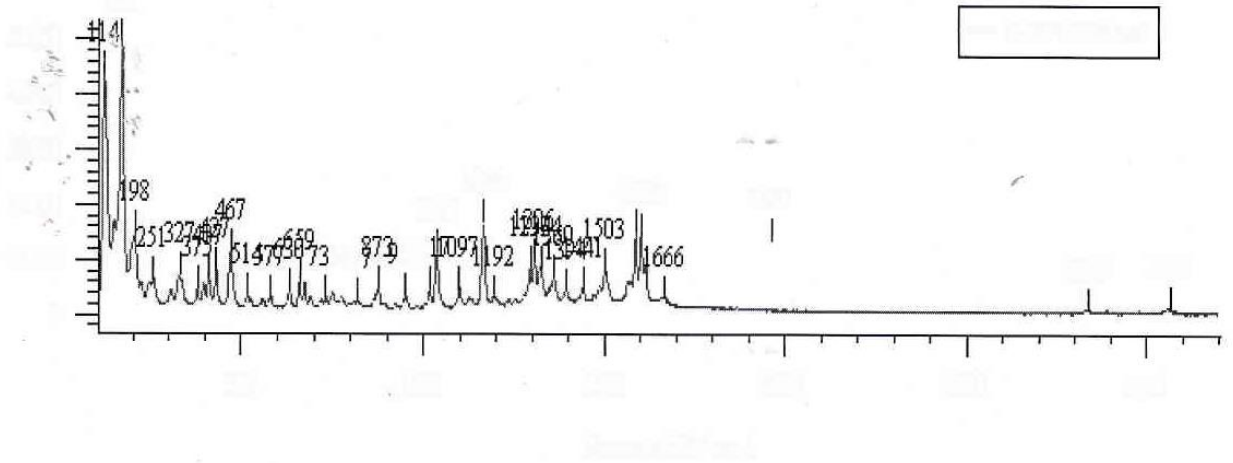


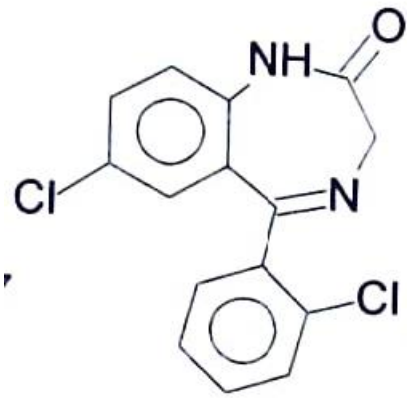
Fig. 5 : Raman spectra of contraband sample

Tables:

Table 1: Rf values of standard and samples.

Sr. No	Standard or control/ Sample	Rf
1	Std. Methaqualone	0.99
2	Std Lorazepam	0.72
3	Std Diazepam	0.98
4	Std Alprazolam	0.65
5	Std Cocaine	0.74
6	Std Ketamine	0.95
7	Std Methamphetamine	0.40
8	Sample	0.90

Table 2 : Raman spectral data of delorazepam.

Sr No	Raman shift cm-1	Assignment	Structure Found
1	427	γ atom ring	 Delorazepam
2	468	C-Cl sretch	
3	631	γ atom ring	
4	660	γC=O	
5	874	γCH	
6	1035	-βCH	
7	1098	-βCH	
8	1164	- βCH	
9	1324	C=C stretch	
10	1359	C=C stretch	
11	1503	NH-def (amide II)	
12	1588	-C=C- stretch	
13	1604	C=O	
14	950	γCH	
15	515	-C—C=O	
16	578	-C-Cl stretch	

17	1394	-C=C- Stretch	
18	731	-CH2	
19	1018	-βCH	
20	1295	C-NH stretch	
21	1307	- βCH	
22	1616	C=N stretch	

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