Technical Note

The Identification of *d-N,N*-Dimethylamphetamine (DMA) in an Exhibit in Malaysia

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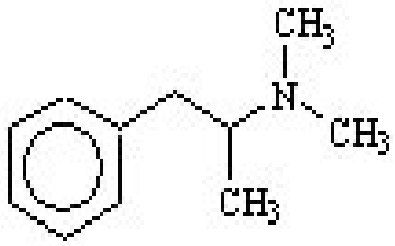
ABSTRACT: A crystalline substance which was suspected to be methamphetamine hydrochloride was instead determined to be *d-N,N*-dimethylamphetamine hydrochloride containing traces of methamphetamine hydrochloride. Analytical data (Color Testing, GC/MS, FTIR, HPLC, Melting Point, Optical Rotation) is reported for *d-N,N*-dimethylamphetamine hydrochloride.

KEYWORDS: *d-N,N*-Dimethylamphetamine Hydrochloride, Color Test, GC-MS, FTIR, HPLC, Melting Point, Optical Rotation, Forensic Chemistry.



Introduction

This laboratory recently received an exhibit consisting of approximately 200 grams of a white crystalline substance that was suspected to be methamphetamine hydrochloride. Crystalline methamphetamine hydrochloride (known locally by the street name of "syabu") is very frequently encountered by the Central Laboratory and its nine branch laboratories. However, in this case the substance was instead determined to be *d-N,N*-dimethylamphetamine hydrochloride with traces of methamphetamine. There have been occasional literature reports of dimethylamphetamine in the United States, some of which included analytical data (*vide infra*); however, those reports were in a law enforcement restricted periodical (*Microgram*), and so are not generally available. More recently, crystalline dimethylamphetamine was reported to be a low prevalence drug of abuse in Japan, making its first appearance there in 1998 [1]. To our knowledge, this is the first report of dimethylamphetamine in Malaysia. Dimethylamphetamine is not currently designated as a controlled substance or "dangerous drug" in Malaysia (that is, like amphetamine or methamphetamine). This paper presents a brief of our analytical findings.

Experimental

Color Test and Reagents

Marquis Reagent and Simon's Regent: These were prepared from analytical grade reagents according to the standard formulations given in the literature [2].

GC/MS

GC/MS analysis was performed on a Shimadzu QP5050A. Column conditions: 30m x 0.25 mm i.d., film thickness 0.25 μm BPX-5 (5% phenylpolysilphenylene-siloxane), with a temperature program starting at 180 ˚C (2 min), then ramping 25 ˚C/min to 250 ˚C. The injection port temperature was 260 ˚C, and the detector and transfer-line temperatures were 280 ˚C.

HPLC

The chromatographic system consisted of a Hewlett Packard Series 1050 HPLC, with a variable UV-detector set at 257 nm and a HP-3396 Series II integrator. The column was a Econosphere (Alltech) 150 mm x 4.6 mm i.d. stainless steel column packed with 5 μm silica. The flow rate was set at 0.8 mL/min. Injections were made via a Rheodyne injection valve with a 20 μL loop. The mobile phase consisted of methanol/water/1N ammonia solution/1N ammonium nitrate (27:3:2:1).

FTIR

Fourier Transfer Infrared Spectroscopy was performed using a Nicolet Magna-IR Spectrometer 550. The resolution was set at 4.000 cm -1, with 32 scans between 4000 cm -1 and 550 cm -1. The sample was determined as a KBr disc.

Melting Point

The melting point was determined using a Buchi B-545 melting point apparatus.

Polarimetry

The optical rotation of two solutions containing 0.048 grams/mL and 0.024 grams/mL of sample in distilled water were measured with a Bellingham & Stanley (London) polarimeter [3]. The accuracy of the instrument was checked by determining the specific optical rotation of a sucrose standard solution (9.78 grams/100mL) and comparing with the literature value [4]. An analytical grade sucrose from Mayer & Baker was used.

Results and Discussion

Color Tests

Treating the sample with the Marquis reagent gave a color change from orange to brown. A faint blue color developed slowly with the Simon's reagent. As a tertiary amine, DMA should not produce a color change with Simon's reagent [5]; therefore, this result suggested the low-level presence of a secondary amine (such as methamphetamine) or some other contaminant.

GC/MS

The GC/MS chromatogram showed two peaks – a small peak preceding a much larger one. The mass spectrum of the large peak (Figure 1) was typical of phenethylamines in that it had a dominant parent ion (at m/z = 72) but otherwise only small fragment ions. The spectrum of the primary component was found to be similar to the literature DMA spectra [6,7,8,9], while the small peak was identified as methamphetamine from its mass spectrum and retention time. The low level presence of methamphetamine was consistent with the findings from the color tests.

HPLC

The HPLC chromatogram also showed two peaks, with a small peak preceding a large peak (Figure 2). The small peak was presumptively identified as methamphetamine from its retention time. The amounts of methamphetamine and DMA were estimated to be 0.5 and 98 percent, respectively, based on the relative peak areas - again, consistent with the GC and color test results.

FTIR

The FTIR spectrum of the sample is shown in Figure 3. This was compared and found to be consistent with the reference IR spectrum of *N,N*-dimethylamphetamine hydrochloride provided by the Forensic Science Laboratory of the Osaka Prefectural Police Headquarters (see Figure 4), and with the spectra given in the literature [6,7,8,9].

Melting Point

The melting point of the sample was found to be 182 -184 ˚C. The melting point of *d*- and *l*- *N,N*-dimethyl-amphetamine hydrochloride is 182-183 ˚C, while racemic *N,N*-dimethylamphetamine has a melting point of 157-159 ˚C (Dr. Munehiro Katagi, Forensic Science Laboratory of the Osaka Prefectural Police Headquarters, personal communication, 2002). This indicated that the sample was either the *d*- or *l*- isomer.

Polarimetry

The sample was purified by recrystallization before being subjected to polarimetry measurements. After three recrystallizations the methamphetamine content was reduced to ca. 0.2 percent (the methamphetamine could not be completely removed). The specific rotation of the purified sample in aqueous medium was determined at two dilutions (0.048 grams/mL and 0.024 grams/mL), and was found to be +14.5 ˚ at 25 ˚C. The sample was thereby identified as *d*-*N,N*-dimethylamphetamine hydrochloride.

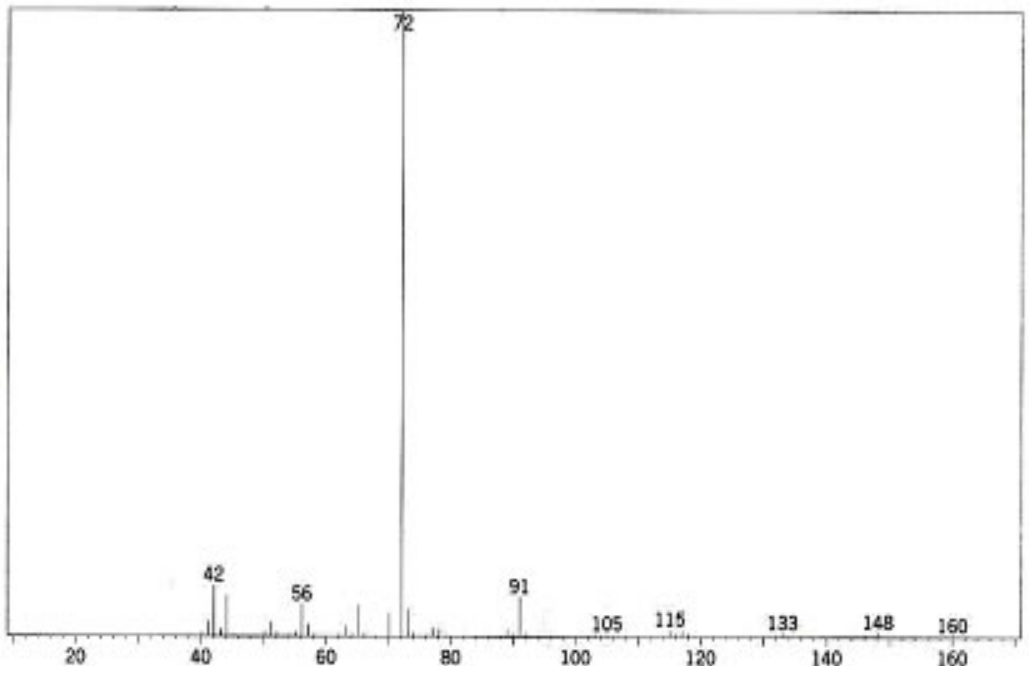


Figure 1. Mass Spectrum of Sample (*N,N*-Dimethylamphetamine)

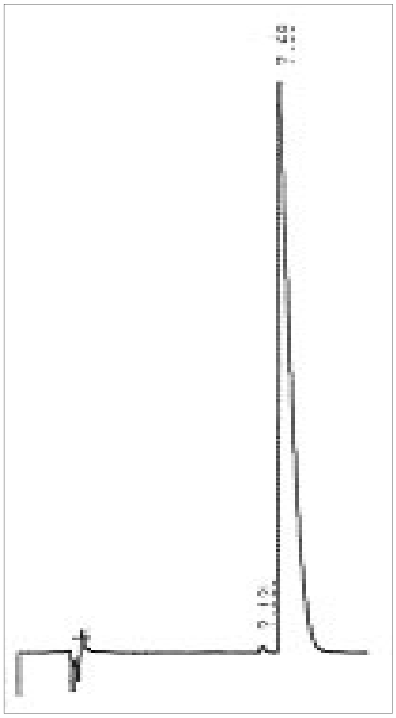


Figure 2. HPLC of Sample (Methamphetamine = 7.12 minutes; *N,N*-Dimethylamphetamine = 7.68 Minutes)

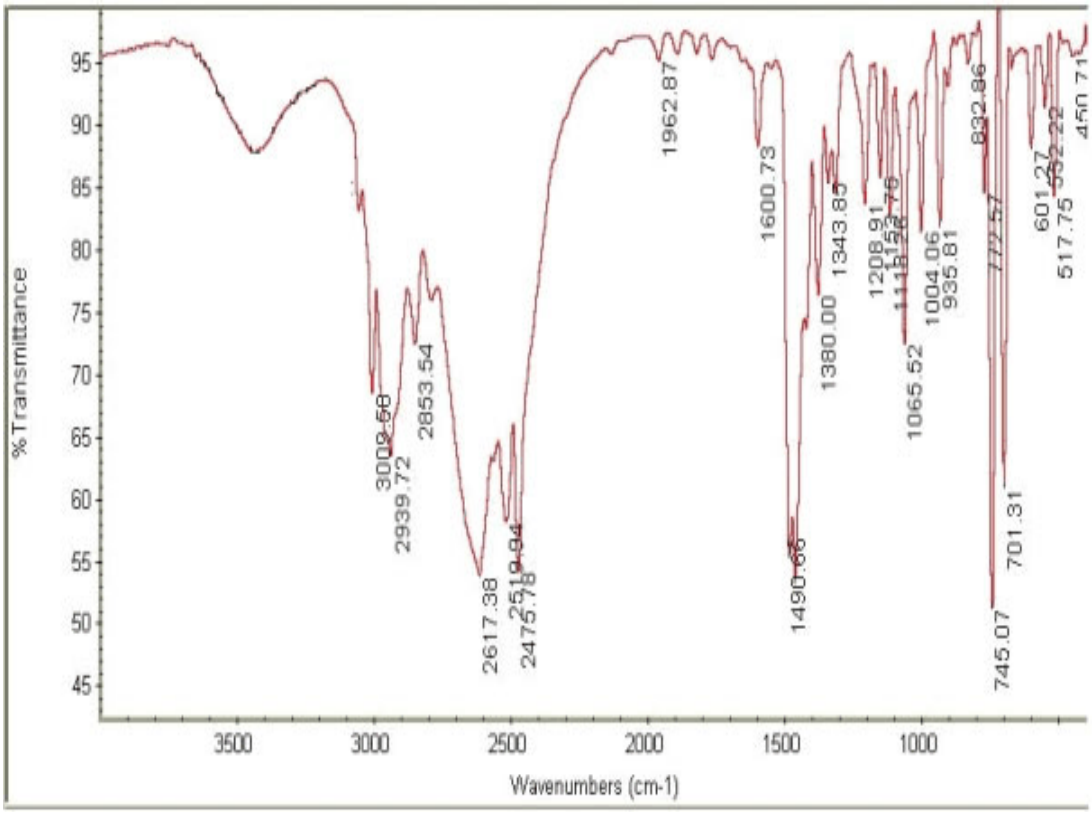


Figure 3: FTIR Spectrum of Sample (98% *N,N*-Dimethylamphetamine Hydrochloride)

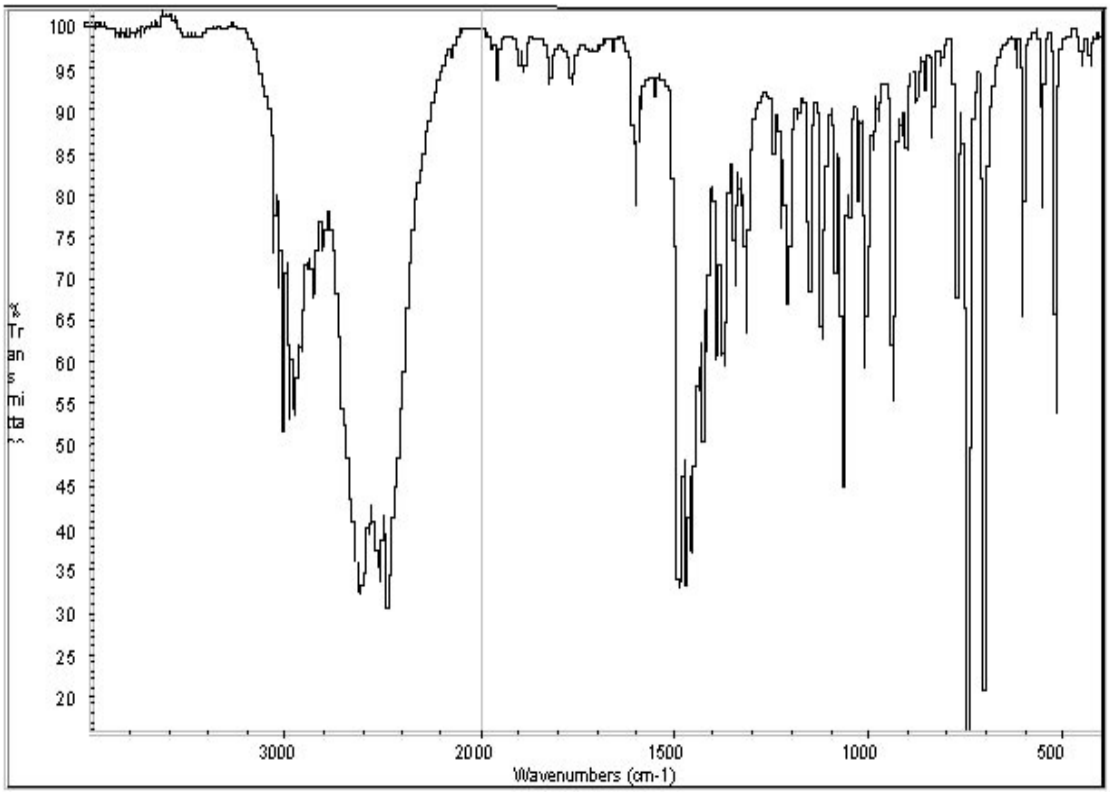


Figure 4: IR Spectrum of Reference Grade *N,N*-Dimethylamphetamine Hydrochloride (Source: Forensic Science Laboratory of the Osaka Prefectural Police Headquarters)

Conclusions

Based on the analytical findings above, the sample was determined to be *d*-*N,N*-dimethylamphetamine hydrochloride contaminated with trace methamphetamine. It is not known whether this sample was synthesized in Malaysia or imported from a neighboring country. There is a remote possibility that the synthesis or importation was done intentionally, since dimethylamphetamine is not a controlled substance in Malaysia. However, if this drug continues to appear in the local illicit drug scene, either in methamphetamine-like crystalline form or mixed with other amphetamine-type stimulants in the form of powders or tablets, then it would likely be eventually included in the list of controlled substances in Malaysia - notwithstanding reports that it exerts much lower CNS stimulant properties versus amphetamine or methamphetamine.[10]

Acknowledgements

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References

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* Note: All issues of *Microgram* (November 1967 - March 2002) and the first nine issues of its successor *Microgram Bulletin* (April - December, 2002) were Law Enforcement Restricted publications, and are therefore unavailable to the general public.

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