

# Seized Blotters Containing One Regioisomer of 25I-NBOMe

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## Abstract

The NBOMe class, mostly represented by the 25I-, 25B- and 25C-NBOMe compounds, is one example among many potentially lethal new psychoactive substances (NPSs) which have emerged in the illegal drugs markets around the world during the last years. Blotters impregnated with NBOMes instead of traditional lysergic acid diethylamide (LSD) have been seized for the first time in mainland China. After proposing the pathway of EI-induced fragmentation of 25I-NBOMe, we also distinguish the possible regioisomers with similar MS results a by the comparison of the fragment ion abundances and the retention time.

**Keywords:** 25I-NBOMe; GC-MS; Lysergic acid diethylamide; Regioisomer

## Introduction

For decades, blotters have been generally impregnated with the potent hallucinogen known as lysergic acid diethylamide (LSD). However, blotters with N-2 methoxybenzyl-substituted phenylethylamine designated as “NBOMes” (or “N-bomb”) have been seized in last years around the world. The NBOMes class, mostly represented by the 25I-, 25B- and 25C-NBOMe compounds, is one example among many potentially lethal new psychoactive substances (NPSs) [1,2]. It is worthy noticed that, according to the previous reports, the deceased may accidentally ingest 25I-NBOMe as counterfeited LSD which is much more dangerous than LSD [3,4]. For this reason, research papers and case reports on these substances are of great interest for the forensic chemistry and toxicology communities [5-7].

Although there were some published reports about the confirmation of these new drugs, the identification of 25I-NBOMe is difficult mainly due to the lack of standards and the proper methodology [8-10]. It is noticeable that there are 3 possible regioisomers with similar MS results (Figure 1). Besides, no experimental case related the confirmation of NBOMes was reported in mainland China to the best of our knowledge.

90 samples of blotters which were suspected of containing LSD were seized last year. We implemented a qualitative method for testing 25-I-NBOMe in blotters by GC-MS, which is applicable to analysis of blotters containing illicit drugs in forensic science laboratories.

## Experiment

### Instrumentation

GC-MS analysis was performed with an Agilent 7890A series gas chromatography system coupled to a 5973 N series mass selective detector manufactured by Agilent (Santa Clara, CA, USA). The extracts were injected automatically in the split mode (20:1) and the injection volume is 1 µL. Chromatographic separation was carried out on a DB-5 capillary column (30 m × 0.25 mm × 0.25 µm) and helium was used as the carrier gas at a constant flow rate of 1 mL/min. The initial column temperature was 60°C, and then increased linearly at a rate of 15°C/min to 300°C, and finally maintained for 15 min to give a total run time of 31 min. The GC injector and the transfer line were maintained at 280°C. The spectrometer was operated in electron impact mode (EI). The temperatures of the ion source and quadrupole were 230°C and 150°C, respectively. Ionization energy was set at 70 eV and positive ions were analyzed. Acquisition was carried out in scan mode from 40 to 500 amu.

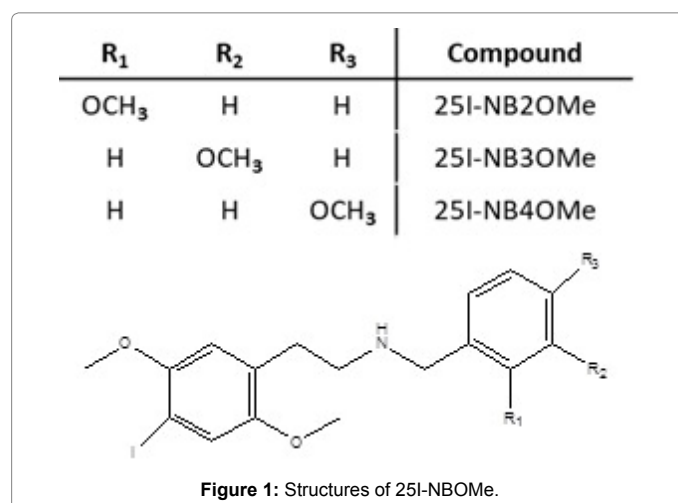


Figure 1: Structures of 25I-NBOMe.

## Materials and reagents

The seized blotters printed with pattern such as the avatar of Marilyn Monroe were investigated (Figure 2). Methanol and chloroform (HPLC grade, purity ≥ 99.9%) were purchased from Fisher Scientific (USA). Analytical grade tartaric acid (purity ≥ 99.5%) and sodium bicarbonate were obtained from sinopharm chemical reagent Co., Ltd (China). Standard LSD and 25I-NBOMe were provided by Sigma-aldrich Company (USA). Deionized water was supplied by reverse diffusion in a Millipore system.

## Sample preparation

Method 1 (named SM1): following the UN recommendations on LSD extraction and testing, four randomly selected stamps (159.14 mg) was soaked in 15 mL 1% tartaric acid and ultrasonicated for 20 min,

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Received February 01, 2018; Accepted February 07, 2018; Published February 14, 2018

Citation: Zhang X, Yanbiao Z, Hongxian Y, Lisheng G Hui Z (2018) Seized Blotters Containing One Regioisomer of 25I-NBOMe. J Forensic Biomed 9: 138. doi: 10.4172/2090-2697.1000138

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**Figure 2:** The blotter papers under investigation.

and then extracted for three times with an equal volume of chloroform. After adjusting the pH of the aqueous phase to be alkaline (pH=8.2) with 1 N NaHCO<sub>3</sub>, 15 mL chloroform was used to extract for three times again. Followed by filtrated and concentrated by centrifugation white crystal was obtained and dissolved by methanol. 1 mL extract was added into a vial for gas chromatography-mass spectroscopy (GC-MS) analysis.

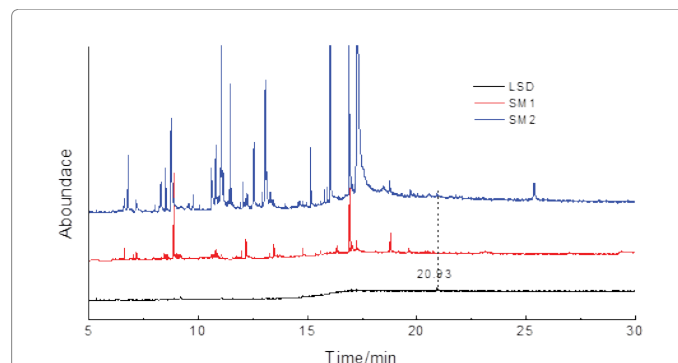
Method 2 (named SM2): Another four randomly selected stamps (158.96 mg) was submerged in 1 mL methanol and extracted for 30 min in an ultrasonic bath. And then the extract was examined by GC-MS.

## Results and Discussion

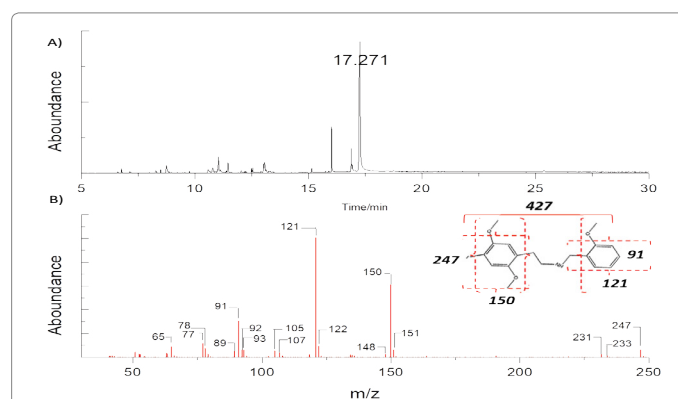
According to the confession of the suspect, the effective drug ingredient was proposed to be LSD. And then the identification of the compound was performance by a comparison of the mass spectrum and retention time of the extract with those of the pure reference material. As shown in Figure 3, no peak corresponding to LSD exists in the total ion chromatogram (TIC) of the extract at the retention time of 20.93 min. By comparing with the curve of 0.002 mg/mL LSD and the extract, the existence of LSD in the investigated blotter was excluded.

Figure 4 shows the GC-MS mass spectrum of the ingredient at the retention time (RT) of 17.27 min. The peaks in the mass spectrum were identified by carefully comparing the underlying mass spectra reported in the literatures [11]. The peak corresponding to the molecular ion, M=427 was contained in the spectrum, though the intensity is very low, which is common in phenethylamines and hinders proper identification of a substance. A relatively intense signal was observed at m/z=396, which is corresponding to M-31 values. Such ions can be formed by the loss of methoxy radical from the molecule [12]. The dominant peak in mass spectrum was observed at m/z=121 resulted from three possibilities including the cleavage of the C-N bond and the C-O bond leading to the formation of 2-methoxybenzylcation [13]. Other peaks at m/z=150, 91, 77 and 65 were also commonly recorded, which were created from common parts of the molecules. The ion observed at m/z=150 could be an iminium cation formed by the dissociation of the C-I bond and the bond between  $\alpha$ - and  $\beta$ -carbon atoms. Such cleavage is a characteristic feature of the mass spectra of phenylethylamines. The decay of the same bond may also lead to dissociation of N-(methoxybenzyl) methylimine molecule and formation of carbocation observed at m/z=277. In turn, the ions with m/z=91, 77 and 65 were characteristic for a benzyl ring containing a methyl substituent. Peak at m/z=91 was a tropylium cation and it was probably formed by the loss of methoxy radical from the predominant ion m/z=121. Noticeably, the relatively intense signals of m/z=277 and 248 corresponding to 150+127 and 121+127 may be resulted from the existence of I. According to the above analysis, 25I-NBOMe was supposed to be contained in the investigated blotters.

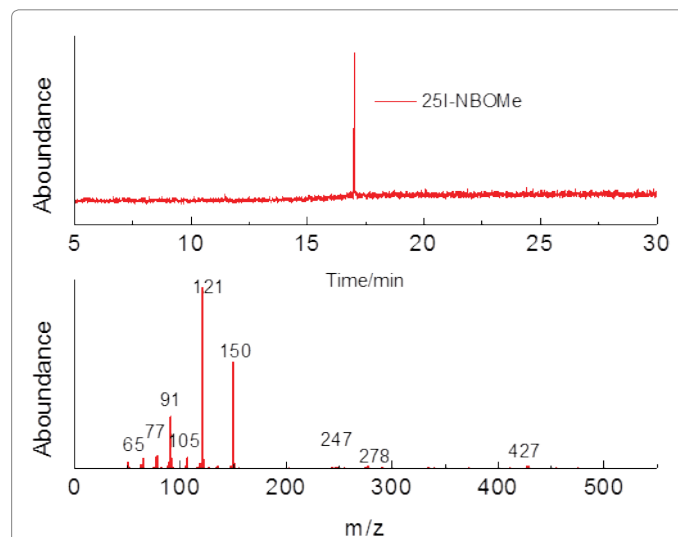
It is noticeable that there are 2 other possible regioisomers with



**Figure 3:** TIC of LSD (0.002 mg/mL) and the blotters extracted by method 1 (SM1) and method 2 (SM2).



**Figure 4:** TIC of SM2 (A) and mass spectrum at the RT of 17.271 min (B).



**Figure 5:** TIC (A) and mass spectrum (B) of 25I-NBOMe.

similar MS results (Figure 1). With this in mind, RT and the ratios of the relative abundance of the fragment ions are very important to certificate the specific structures. For this reason, identification was based upon comparison of the retention time and enhanced product ion scan with reference standards (Figure 5). What's more, the tropylium ion (m/z=91) abundances were 29%, and the relative abundance of the fragment ion produced at m/z 150 is 62%, which is consistency with the previous report [14].

## Conclusion

In this report, the seized blotters contained a class of new psychoactive substance were investigated for the first time showing the evolution of NBOMes in mainland China. Moreover, based on the developed and implemented qualitative method for testing 25-I-NBOMe in blotters by GC-MS, the pathway of EI-induced fragmentation of 25-I-NBOMe was proposed. Additionally, it is worthy for antinarcotics departments to note that NBOMes are becoming potential hallucinogenic drugs consumed in China.

## Acknowledgement

We appreciate the support from the Basic Research Program Exclusive for Central Government-Class Institute of Public Welfare (No.2018JB026).

## Conflict of Interest

There is no conflict of interest.

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