— Minireview —

Update on Clandestine Amphetamines and Their Analogues Recently Seen in Japan

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Amphetamines and their analogues have undergone a cycle of popularity as recreational drugs in Japan. The current wave of popularity began in the early 1990s and spread throughout the country. More recently, not only 3,4-methylenedioxymethamphetamine (MDMA) but also other designer amphetamines analogues, including *p*-methoxyamphetamine (PMA), 2,5-dimethoxy-4-ethylthiophenethylamine (2C-T-2), *etc*., have been extensively and increasingly abused, mainly among juveniles. This minireview presents an update on the amphetamines and their analogues by focusing on clandestine tablets encountered in Japan recently.

Key words —— amphetamines, amphetamine analogue, clandestine tablet

INTRODUCTION

Drug abuse, which affects human nature and causes numerous crimes, has become a serious problem throughout the world. *d*-Methamphetamine hydrochloride (*d*-MA HCl) crystalline has been the most extensively and increasingly used illicit drug in Japan, although *l*-MA HCl crystalline has also often been encountered in the last four autumn seasons.[1–3] Additionally, *d*-dimethylamphetamine hydrochloride (*d*-DMA HCl) crystalline emerged in 1998 and can sometimes be seen even now.[4,5]

More recently, the abuse of other drugs, including amphetamine analogues, has also been on the rise. Especially, many kinds of illicitly manufactured tablets containing amphetamines and their analogues are becoming widespread among juveniles because they are not only fashionable but also easy to get, for instance, *via* the internet, and to carry and use without injection.

There are hundreds of possible amphetamine analogues that modify the basic amphetamine structure and retain or modify the stimulant effects of a parent compound (Fig. 1), making it difficult to identify them. A number of analytical procedures for identification of amphetamines and their analogues have recently been reported.[6–10]

This minireview presents an update on amphetamines and their analogues which have been submitted to our laboratory for forensic drug analysis primarily in tablet form.

Amphetamines

In most cases, amphetamines contained in clandestine tablets seized in Japan have been found to be MA. Well known by the name of "Yāba," the tablets were illicitly manufactured, for the most part, in the "Golden Triangle" and usually had a colorful color and some kind of logo such as "wy" and "66 (or 99)"on one or both sides (Table 1). Most of them were found to contain MA ranging from 8.9 to 14 mg per tablet by gas chromatography-mass spectrometry (GC-MS) and high-performance liquid chromatography-mass spectrometry (LC-MS) determination in our laboratory. They have also often been confirmed to contain other pharmaceutical agents as additives (*e.g*., caffeine, theophylline and ketamine).

In rare cases, amphetamine (AP) tablets were encountered in 1998 and contained AP as their sole or primary ingredient. Some of them also contained smaller amounts of caffeine, diacetylmorphine, 6-acetylmorphine and opium components, including morphine, papaverine, noscapine and meconic acid. All of them were found to contained phenylacetone and *N*-formyl amphetamine, which were thought to

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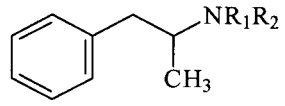
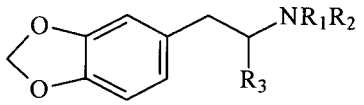
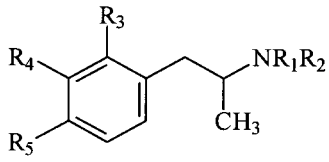


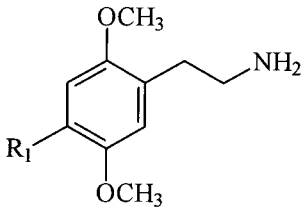
AP; R_1 = H and R_2 = H
MA; R_1 = H and R_2 = CH_3
DMA; R_1 = CH_3 and R_2 = CH_3



MDA; R_1 = H, R_2 = H, and R_3 = CH_3
MDMA; R_1 = H, R_2 = CH_3, and R_3 = CH_3
MDEA; R_1 = H, R_2 = C_2H_5, and R_3 = CH_3
MBDB; R_1 = H, R_2 = CH_3, and R_3 = C_2H_5



OMA; R_1 = H, R_2 = H, R_3 = OCH_3, R_4 = H, and R_5 = H
MMA; R_1 = H, R_2 = H, R_3 = H, R_4 = OCH_3, and R_5 = H
PMA; R_1 = H, R_2 = H, R_3 = H, R_4 = H, and R_5 = OCH_3
OMMA; R_1 = H, R_2 = CH_3, R_3 = OCH_3, R_4 = H, and R_5 = H
MMMA; R_1 = H, R_2 = CH_3, R_3 = H, R_4 = OCH_3, and R_5 = H
PMMA; R_1 = H, R_2 = CH_3, R_3 = H, R_4 = H, and R_5 = OCH_3
OMDM; R_1 = CH_3, R_2 = CH_3, R_3 = OCH_3, R_4 = H, and R_5 = H
MMDM; R_1 = CH_3, R_2 = CH_3, R_3 = H, R_4 = OCH_3, and R_5 = H
PMDM; R_1 = CH_3, R_2 = CH_3, R_3 = H, R_4 = H, and R_5 = OCH_3



2C-B; R_1 =Br
2C-T-2; R_1 = C_2H_5S
2C-T-7; R_1 = *n*-C_3H_7S

Fig. 1. Chemical Structures of Some Amphetamines and Their Analogs

Table 1. Clandestine Amphetamines Tablets Encountered in Osaka

LOGO	Color	Diameter	Weight	Top shape	Side view	Scoring	Active ingredients[a)]
66 (or 99)	purple	6.5 mm	100 mg	round	flat/convex	half score	MA 8.9 mg Caffeine
66 (or 99)/ M (or W)	purple	7.1 mm	100 mg	round	biconvex	no score	MA 14 mg Caffeine Theophylline
wy	orange	6.0 mm	90 mg	round	biconvex	no score	MA 10 mg Caffeine
P	green mottled	8.3 mm	370 mg	round	flat/beveled	no score	MA 8.9 mg Ketamine 200 mg Diazepam Chlorpromazine
[turtle]	red mottled	8.1 mm	280 mg	round	flat/beveled	no score	AP 46 mg AP 26 mg HER 0.11 mg 6-AM 0.51 mg MOR 0.095 mg Papaverine Noscapine Meconic acid Caffeine, *etc.*
[unknown]/199	white	10.5 mm	280 mg	round	biconvex	half score	MA 0.12 mg Yohimbine

a) Quantitation was performed on a LCMS-QP8000 (Shimadzu) equipped with an electrospray ionization interface by selected ion monitoring (SIM) mode. The values expressed were calculated as the free base.

Table 2. Clandestine Amphetamine Analogues Tablets Encountered in Osaka

LOGO	Color	Diameter	Weight	Top shape	Side view	Scoring	Active ingredients
Y2K	off-white	9.2 mm	290 mg	round	flat/beveled	no score	MDMA 73 mg AP 4.4 mg
Y2K	light green and blue mottled	9.2 mm	230 mg	round	flat no beveled	no score	MDMA 87 mg MA 30 mg
RN	off-white	8.4 mm	250 mg	round	flat/convex	half score	MDMA 83 mg AP 9.3 mg
RN	light green	9.2 mm	300 mg	round	flat/beveled	half score	MDMA 120 mg
RN	green mottled	8.1 mm	300 mg	round	flat/beveled	Half score	MDMA 120 mg
JB	light blue and purple mottled	8.4 mm	290 mg	round	biconvex	no score	MDMA 62 mg
M3	yellow	8.1 mm	310 mg	round	flat/beveled	no score	MDMA 130 mg
[smiling sun]	off-white	9.3 mm	290 mg	round	flat /beveled	half score	MDMA 160 mg
SKY	green mottled	8.1 mm	310 mg	round	flat/convex	half score	MDMA 100 mg
[“KAPPA” logo]	light green	9.3 mm	320 mg	round	flat /beveled	half score	MDMA 81 mg
[none]	light brown mottled	8.4 mm	320 mg	round	biconvex	no score	MDMA 120 mg
[none]	off-white	7.2 mm	290 mg	round	flat/beveled	half score	MDMA 88 mg
[none]	red mottled	6.9 mm	140 mg	round	biconvex	no score	MDMA 64 mg
[“Mitsubishi” logo]	off-white	8.1 mm	340 mg	round	biconvex	half score	MDMA 65 mg
[“Mitsubishi” logo]	off-white	8.2 mm	340 mg	round	flat no beveled	half score	MDMA 98 mg
[“Mitsubishi” logo]	off-white	9.1 mm	340 mg	round	biconex	half score	MDMA 130 mg
[“Chanel” logo]	pink	8.2 mm	250 mg	round	flat/beveled	no score	MDMA 89 mg MA 1.0 mg AP 6.9 mg Caffeine Ketamine
○	light green mottled	8.2 mm	270 mg	round	flat/beveled	no score	MDMA 90 mg

be the raw material and an intermediate of AP, respectively.[11)]

Amphetamine Analogues

1) 3,4-Methylenedioxyamphetamines (MDAs)

The MDAs are a new drug class, “entactogens,” which enhance understanding, communicativeness and empathy almost without showing hallucinogenic effects.[12–14)] They include 3,4-methylenedioxyamphetamine (MDA), 3,4-methylenedioxymethamphetamine (MDMA), 3,4-methylenedioxyethylamphetamine (MDEA), *N*-methyl-1-(3,4-methylenedioxyphenyl)-2-butanamine (MBDB), *etc*., and all but MDA, MDMA, and MDEA are not currently classified as illegal drugs.

Among these drugs, MDMA is now the most popular recreational drug in Japan. It was first synthesized in 1914 for use as an appetite suppressant. It emerged as a major recreational drug at the end of the 1960s in the U.S.A. and in the middle of the 1980s in Europe, and is now becoming even more popular in the former. It is well known by the street name of “Ecstasy,” and is usually sold in the form of tablets in Japan. The tablets often have various logos, including the 3-diamond (“Mitsubishi” mark), “Y2K” or “RN,” *etc.,* on one or both sides (Table 2). Using GC-MS and LC-MS determination in our laboratory, they were found to contain various amounts of MDMA ranging from 28 to 160 mg per tablet as the primary ingredient, and sometimes a smaller amount of amphetamines or/and other pharmaceutical agents (*e.g*., caffeine, ketamine). They also have sometimes contained other MDAs such as MDA or MDEA (Table 2).

2) *p*-Methoxyamphetamines (PMAs)

More recently in 2001, PMAs tablets have been encountered in Osaka and Aichi Prefectures. They carried the 3-diamond “Mitsubishi” logo and were sold as Ecstasy, MDMA. GC-MS and nuclear mag-

Table 2. Continued

LOGO	Color	Diameter	Weight	Top shape	Side view	Scoring	Active ingredients
[none]	blue mottled	8.4 mm	330 mg	round	flat/beveled	no score	MDMA 110 mg MA 1.7 mg Ketamine
[sparrow]	off-white	9.1 mm	270 mg	round	flat/beveled	half score	MDMA 28 mg MDEA 49 mg
[“Mitsubishi” logo] /[monster face]	off-white	8.3 mm	250 mg	round	flat/beveled	no score	MDA 86 mg
[“Mitsubishi” logo] (both sides)	light brown mottled	8.3 mm	250 mg	round	flat/beveled	no score	MDA 100 mg
[“Mitsubishi” logo] /[lips]	light brown mottled	8.2 mm	250 mg	round	flat/beveled	no score	MDA 94 mg
[“Mitsubishi” logo]	off-white	7.1 mm	220 mg	round	flat/beveled	no score	PMA 51 mg PMMA 17mg AP 17 mg Ephedrine
	light beige mottled						PMA 55 mg PMMA 17 mg PMDM (trace) Ephedrine
							PMA 62 mg PMMA 8.6 mg PMDM 7.0 mg Ephedrine
[none]	white	5.1 mm	46 mg	round	flat/beveled	no score	2C-B (no quant)

a) Quantitation was performed on a LCMS-QP8000 equipped with an electrospray ionization interface by selected ion monitoring (SIM) mode. The values expressed were calculated as the free base.

netic resonance spectroscopy (NMR) in our laboratory revealed that the tablets did not contain MDMA, but rather PMA, *p*-methoxymethamphetamine (PMMA) or *p*-methoxydimethylamphetamine (PMDM), or even some combination of the three as primary ingredient (Table 2).

Among these, only PMA designated as a controlled drug, while others are not controlled. It is well known that PMA has a hallucinogenic property which is about 5 times more potent than mescaline,[15] though it is reported that the physiological effects of PMA and PMMA are markedly different: PMMA has MDMA-like effects whereas PMA has a degree of an AP-like character, but no MDMA-like character.[16–18] PMA is routinely sold as “Ecstasy.” However, it lacks the MDMA effect but is more toxic, leading to overdosing and even to death in pursuit of the same effect. Indeed, 9 deaths in Canada associated with PMA were reported in 1973,[19] and 16 deaths have been reported in Australia since 1994.[20,21] Widespread abuse of PMA and PMMA in Spain was also reported in the mid-1990s.[22] More recently, the U.S. Drug Enforcement Administration (DEA) has also encountered some tablets with the Mitsubishi 3-diamond logo that contain PMAs, which have been linked to several deaths there.[23]

For metabolism and excretion of PMA in human, Kitchen et al. reported that an average of 15% was eliminated as unchanged drug, 18% as free *p*-hydroxyamphetamine, 21% as conjugated *p*-hydroxyamphetamine, 7% as conjugated *N*-hydroxy-PMA, and 4% as *p*-hydroxynorephedrine.[24]

There is some analytical data available on PMAs, including color tests, GC-MS, infrared spectroscopy (IR), *etc*.,[23,25–28] but it is difficult to differentiate PMAs from other corresponding ortho- and meta-isomers of methoxyamphetamines (MAPs). Mass spectra of PMAs, in particular, are almost the same as those of their corresponding isomers, and examination of their NMR spectra is therefore required for unequivocal identification.

To discriminate these isomers, we prepared each isomer of methoxyamphetamine, methoxymeth-

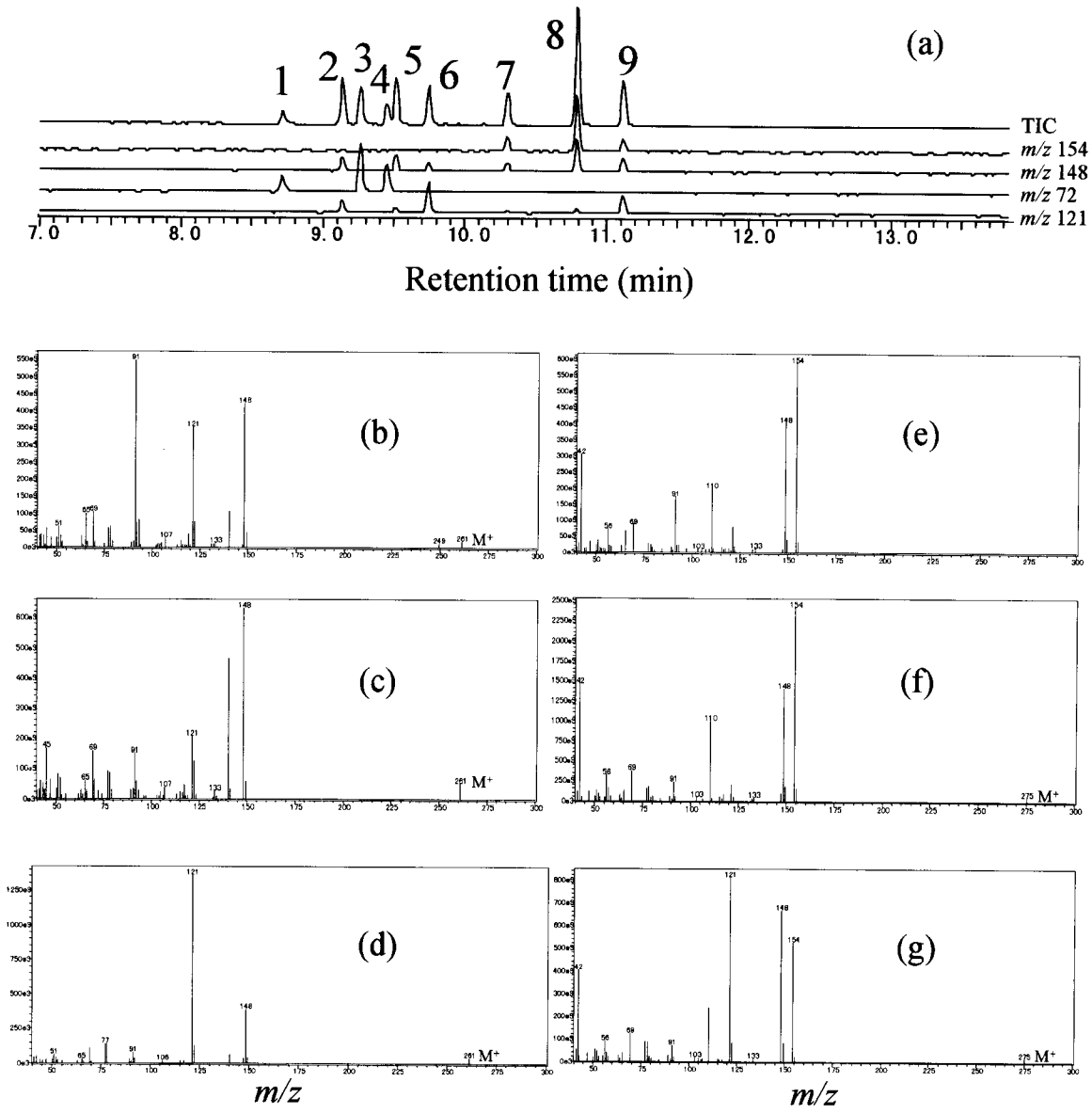


Fig. 2. (a) Total Ion Chromatogram and Extracted Ion Chromatograms Obtained for Trifluoroacetyl (TFA) Derivatives of Authentic Methoxyamphetamines, and EI Mass Spectra of TFA Derivatives of (b) OMA, (c) MMA, (d) PMA, (e) OMMA, (f) MMMA, and (g) PMMA

Methoxydimethylamphetamines cannot be derivatized, and their mass spectra are omitted.Peaks: 1, *o*-methoxydimethylamphetamine (OMDM); 2, *o*-methoxyamphetamine (OMA); 3, *m*-methoxydimethyamphetamine (MMDM); 4, *p*-PMDM; 5, *m*-methoxyamphetamine (MMA); 6, *p*-methoxyamphetamine (PMA); 7, *o*-methoxymethamphetamine (OMMA); 8, *m*-methoxymethamphetamine (MMMA); 9, *p*- PMMA. GC-MS was performed on QP5050 (Shimadzu, Kyoto, Japan). Column conditions: 30 m × 32 mm i.d. DB-1, helium flow rate of 3 ml/min, temperature program of 70°C (1 min), 15°C/min to 250°C.

amphetamine and methoxydimethylamphetamine by reductive amination of orto-, meta-, and para-methoxy phenylacetone using the appropriate amine hydrochloride and sodium cyanoborohydride, and performed GC-MS analysis after trifluoroacetyl (TFA) derivatization of PMAs. We successfully identified with their mass spectral information except for isomers of methoxydimethylamphetamine as shown in Fig. 2; when differentiating isomers of methoxydimethylamphetamines, their retention times are very effective.

3) Others

2,5-Dimethoxyphenalkylamines have recently been encountered in Japan. They include 4-bromo-2,5-dimethoxyphenethylamine (2C-B), 2,5-dimethoxy-4-ethylthiophenethylamine (2C-T-2), and 2,5-dimethoxy-4-(*n*)-propylthiophenethylamine

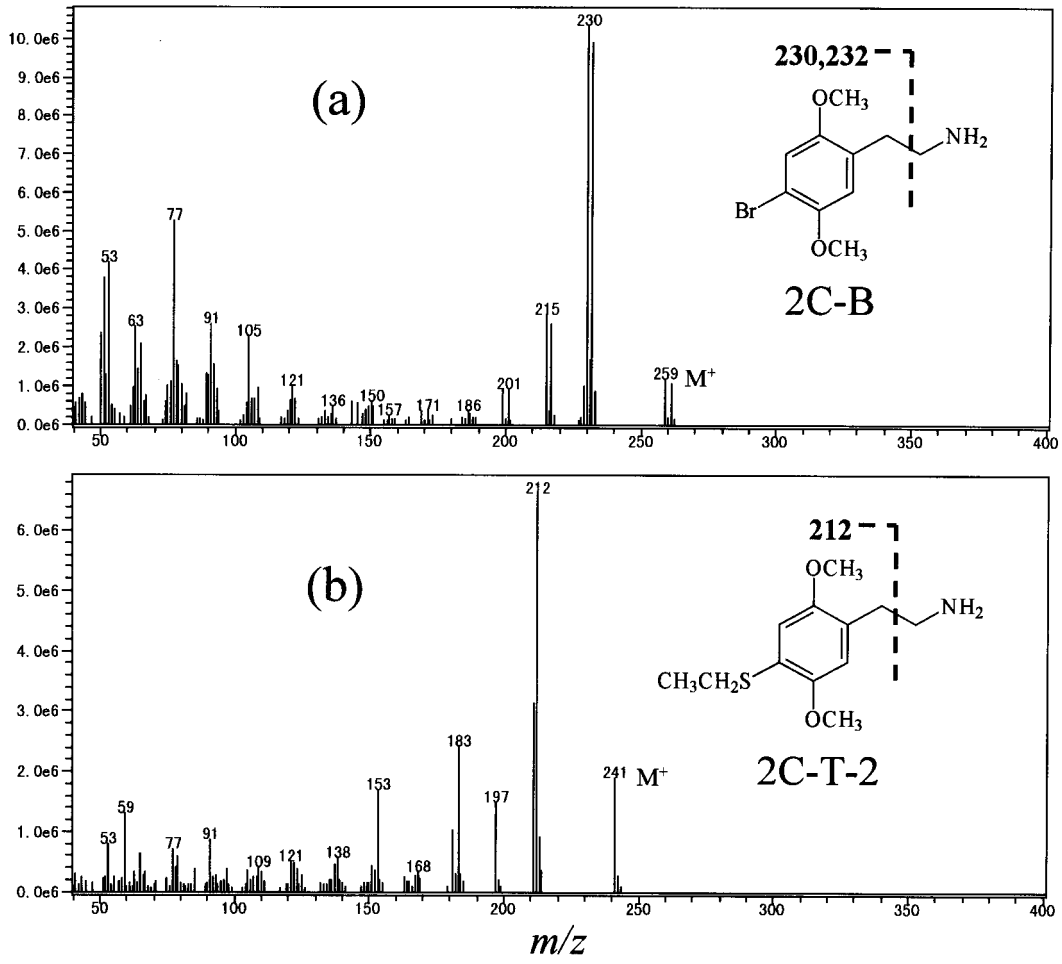


Fig. 3. EI Mass Spectra of (a) 2C-B and (b) 2C-T-2
Mass spectra were measured by GC-MS on GCMS-QP5050 (Shimadzu).

(2C-T-7). They are allegedly manufactured in the Netherlands, and are sold in Japan as new hallucinogenic drugs mainly via the internet. 2C-B (street name "Nexus," "Venus," "Bromo," *etc*.) is known to show a psychotropic effect at much lower dosages (4–30 mg) than MDMA, and that higher doses may cause frightening hallucination and unexpected heavy "trips."[29,30] It emerged as a hallucinogenic drug on the Dutch market in 1995, and became quite popular in a short period in Europe. It was therefore placed on the list of illegal drugs in the Netherlands in 1997, and in Japan in 1998.

These measures prevented further popularity of 2C-B. However, new 2,5-dimethoxyphenalkylamine drugs, 2C-T-2 and 2C-T-7 were recently introduced on the black market as substitute hallucinogenic drugs for 2C-B in Japan as well as in Europe and U.S.A. These are not currently classified as illegal drugs, and nothing prevents their sale and abuse; there is thus much apprehension of their popularity in Japan.

For identification of these substances, some of the data from color tests, IR, NMR, GC-MS are available.[30–32] Especially, their mass spectra give molecular ions M^+ and some specific fragment ions as depicted in Fig. 3, and GC-MS analysis is very effective; but their formal identification is difficult because their positional isomers can be found.[30] For unequivocal identification of these substances in seized exhibits, their basic structure must be recognized (for example dimethoxyethylthiophenethylamine backbone for 2C-T-2) by GC-MS, and the complete substitution pattern of the benzenic ring assigned by NMR. Indeed, 2C-T-2 was identified in seized tan powder by GC-MS and NMR at our laboratory.

With regard to their pharmacological properties, metabolism and toxicity, only scarce data exist,[29,33] and future studies on these subjects are required.

5-Methoxy-*N*,*N*-dimethyltryptamine which has

high hallucinogenic activity[34] has been encountered by our laboratory, and benzylpiperazine which has an amphetamine-like effect[35] have also been seen recently in Hyogo Prefecture. The standards of both compounds are commercially available, and no analytical data is shown here.

CONCLUSION

An update on amphetamines and their analogues seen recently in Japan has been presented. Modification to a basic amphetamine structure can easily produce new designer amphetamines and their analogues, and these have indeed been manufactured and increasingly introduced on the Japanese market *via* smuggling and the internet. The diversity of the compounds results in difficulty in their identification and in recognizing what they are. Thus, in the forensic drug analysis, it is necessary to make up-to-date analytical data and reference compounds available and to unequivocally identify the compounds using the analytical tools of mass spectrometry, NMR, and IR.

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