

Profiling of Ecstasy Tablets Seized in Japan

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ABSTRACT: 54 Ecstasy tablets seized in Japan during the first half of CY-2002 were analyzed to determine their physical and chemical characteristics and to derive a “snapshot” comparison with seizures made in CY’s 2000 and 2001. For physical characterization, logo, cleavage, coat, vertical view, horizontal view, diameter, thickness, weight, smell, outside color, inside color, color, toughness, capping, and logo were measured, and a photograph was taken. For chemical characterization, the tablet components were identified by GC/MS and HPLC, with quantification by HPLC. The maximum content of 3,4-methylenedioxymethamphetamine was 160 milligrams/tablet. Other tablet components detected were 3,4-methylenedioxyamphetamine, ephedrine, caffeine, ketamine, and methamphetamine. Several trends in the chemical characteristics are presented.

KEYWORDS: 3,4-Methylenedioxymethamphetamine, MDMA, Ecstasy, Characterization, Profiling, Ketamine, Forensic Chemistry.

Introduction

Abuse of 3,4-methylenedioxymethamphetamine (“Ecstasy”) has spread worldwide. Although still a very small percentage relative to worldwide consumption, the number of Ecstasy tablets seized in Japan has been rapidly increasing, with 174,000 tablets seized in 2002 [1]. Ecstasy abuse in Japan is considered to be a very serious problem, similar to methamphetamine abuse. We recently profiled Ecstasy tablets seized in Japan in CY’s 2000 and 2001, and reported the results in the *Journal of Health Science* [2]. We have continued to profile Ecstasy tablets using the same methodologies presented in the *Journal of Health Science’s* article [2]. Herein, we present the results of the profiling of Ecstasy tablets seized in Japan during the first half of CY-2002.

Experimental

Chemicals

Methamphetamine hydrochloride, ephedrine hydrochloride, and caffeine were obtained from commercial sources in Japan. 3,4-Methylenedioxyamphetamine hydrochloride (MDA), 3,4-methylenedioxymethamphetamine hydrochloride (MDMA), and 3,4-methylenedioxyethylamphetamine hydrochloride (MDEA) were obtained from the reference collection of the Narcotics Control Department Laboratory. All solvents were of HPLC grade. The 54 tablets that were analyzed in the present study were randomly selected from among seizures forfeited to the Government during the first half of CY-2002.

Physical Characteristics

Fifteen parameters (logo, cleavage, coat, vertical view, horizontal view, diameter, thickness, weight, smell, outside color, inside color, color, toughness, capping, and appearance of logo) were measured to establish the

physical characteristics of each tablet. The tablets were also photographed with a digital camera. An identification number was applied to each tablet, consisting of a logo number, color number, and serial number.

Extraction Procedure

Each tablet was placed in an agate mortar and crushed to a fine powder. A 10 mg portion of the resulting powder was dissolved in 8 mL of phosphate buffer (pH 7.0) by shaking for 5 minutes. The solution was centrifuged for 10 minutes at 3,500 rpm, and 100 μ L of the supernatant liquid was transferred to a small autosampler vial for HPLC analysis. Half of the remaining solution (4 mL) was used for the identification of the basic compounds (e.g., ephedrine, MDA, MDMA, etc.), while the other half was used for the identification of the neutral compounds (i.e., caffeine). One mL of 10% Na₂CO₃ solution was added to adjust the first 4 mL aliquot of solution to pH 10.5, and the mixture was then extracted with 3 mL of chloroform by shaking for 5 minutes. The biphasic solution was then centrifuged at 3,500 rpm for 10 minutes, after which the organic layer was transferred to a vial for GC/MS analysis. The second 4 mL aliquot of solution (still at pH 7.0) was similarly extracted with 3 mL of chloroform, for identification of caffeine by GC/MS.

GC/MS Analysis

A GC/MS equipped with a Hewlett-Packard (HP) 6890 Series Gas Chromatograph, a double-focusing mass spectrometer Mstation (JEOL, Tokyo, Japan), and a data processing XMS system (JEOL, Tokyo, Japan), were used for identification of the components in the tablets. An Ultra-2 fused-silica capillary column (30 m x 0.2 mm with 0.33 μ m HP) was inserted directly into the ion source of the mass spectrometer, and analysis was performed in the splitless mode with Helium as the carrier gas. The GC temperature programming was run from 50 °C (1 minute) to 300 °C (4 minutes) at 10 °C /minute, with the injection port at 250 °C. Electron-impact ionization mass conditions were set as follows: Ionizing energy, 70 eV; ionization current, 300 μ A; and ion-source temperature, 300 °C. Mass spectra were obtained using the scanning mode.

HPLC Analysis

A Shiseido Nanospace HPLC, equipped with a UV detector linked to a data system (S-MC, Shiseido, Tokyo, Japan), was used for qualitative and quantitative analysis of the components in the tablets. Chromatographic separation was achieved using an ODS-type semi-microcolumn (CAPCELL PAK C18 UG 120 S5, 250 mm x 1.5 mm i.d.). The mobile phase used for ephedrine, MDA, MDMA, MDEA, methamphetamine, and ketamine was 5 mmol/L SDS in 20 mmol/L KH₂PO₄-CH₃CN (65:35). The mobile phase used for caffeine was H₂O-methanol (7:3). The flow rate was maintained at 0.1 mL/minute. Separation was carried out at 50 °C for ephedrine, MDA, MDMA, MDEA, methamphetamine, and ketamine, and 35 °C for caffeine. The monitoring wavelength was 210 nm for ephedrine, MDA, MDMA, MDEA, methamphetamine, and ketamine, and 254 nm for caffeine. Good linearity for this quantitative analysis was confirmed over the concentration range of 0.1 – 0.8 mg/mL ($r^2 = 0.9993 - 0.9997$ for six compounds).

Results and Discussion

Physical Characteristics

To date, there are few reports concerning the physical or chemical characteristics of Ecstasy tablets in Japan [3,4]. To aid in quick comparison, full-color photographs of all tablets are shown in order of the amount of MDMA or MDA as an active ingredient, followed by a group containing mixed drugs (Figure 1). The characteristic physical properties are listed in Table 1. The diameters ranged from 7.1 – 10.1 mm, the widths ranged from 2.6 – 7.0 mm, and the weights ranged from 105 – 348 mg.

















Chemical Characteristics





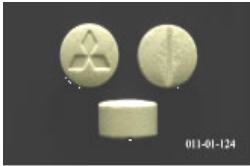















The active ingredients in each tablet were identified by GC/MS and HPLC. The detected components were MDMA, MDA, ephedrine, caffeine, ketamine, and methamphetamine. Thirty-five tablets contained MDMA as the sole active ingredient. The content range (calculated as MDMA hydrochloride) was 37 - 160 mg/tablet. One tablet contained MDA alone. The content was calculated as MDA hydrochloride at 75 mg/tablet. Eighteen tablets contained two or three active ingredients. To summarize the findings, we have noted the following trends as compared with last report [2]:

















1. An increase in tablets containing ketamine.
2. An increase in tablets containing methamphetamine.
3. A decrease in tablets containing ephedrine.

We are continuing to profile Ecstasy tablets seized in Japan [4].

Figure 1. Photographs of 54 Ecstasy Tablets Seized in Japan During the First Half of CY-2002, with Logo Name and Amount of Active Ingredient.

| | | | |
|--|---|---|--|
| <p>Tulip</p>  <p>MDMA 160 mg/tab</p> | <p>B29</p>  <p>MDMA 157 mg/tab</p> | <p>B29</p>  <p>MDMA 151 mg/tab</p> | <p>XL</p>  <p>MDMA 146 mg/tab</p> |
| <p>Mitsubishi</p>  <p>MDMA 103 mg/tab</p> | <p>cK</p>  <p>MDMA 98 mg/tab</p> | <p>XL</p>  <p>MDMA 98mg/tab</p> | <p>Bird (Fry)</p>  <p>MDMA 89 mg/tab</p> |
| <p>Mickey Mouse</p>  <p>MDMA 88 mg/tab</p> | <p>Crocodile</p>  <p>MDMA 87 mg/tab</p> | <p>CU</p>  <p>MDMA 87 mg/tab</p> | <p>FF</p>  <p>MDMA 80 mg/tab</p> |
| <p>Star</p>  <p>MDMA 77 mg/tab</p> | <p>007</p>  <p>MDMA 70 mg/tab</p> | <p>Smiley</p>  <p>MDMA 69 mg/tab</p> | <p>(No Logo)</p>  <p>MDMA 66 mg/tab</p> |

| | | | |
|--|---|---|---|
| <p>Mitsubishi</p>  <p>MDMA 64 mg/tab</p> | <p>!</p>  <p>MDMA 64 mg/tab</p> | <p>Mitsubishi</p>  <p>MDMA 64 mg/tab</p> | <p>Butterfly</p>  <p>MDMA 63 mg/tab</p> |
| <p>Mitsubishi</p>  <p>MDMA 61 mg/tab</p> | <p>Lozenge</p>  <p>MDMA 61 mg/tab</p> | <p>Superman</p>  <p>MDMA 60 mg/tab</p> | <p>Armani</p>  <p>MDMA 59 mg/tab</p> |
| <p>Lozenge</p>  <p>MDMA 57 mg/tab</p> | <p>Propeller</p>  <p>MDMA 57 mg/tab</p> | <p>Cellular Phone</p>  <p>MDMA 56 mg/tab</p> | <p>Smurf</p>  <p>MDMA 54 mg/tab</p> |
| <p>Motorola</p>  <p>MDMA 51 mg/tab</p> | <p>007</p>  <p>MDMA 50 mg/tab</p> | <p>Smiley</p>  <p>MDMA 46 mg/tab</p> | <p>Fish</p>  <p>MDMA 42 mg/tab</p> |
| <p>Hole</p>  <p>MDMA 42 mg/tab</p> | <p>Smiley</p>  <p>MDMA 40 mg/tab</p> | <p>Mercedes</p>  <p>MDMA 37 mg/tab</p> | <p>Tower</p>  <p>MDMA 106 mg/tab Caffeine 35 mg/tab</p> |

| | | | |
|--|--|---|--|
| <p style="text-align: center;">Tower</p>  <p style="text-align: right; font-size: small;">049-13-108</p> <p style="text-align: center;">MDMA 98 mg/tab Caffeine 29 mg/tab</p> | <p style="text-align: center;">Popeye</p>  <p style="text-align: right; font-size: small;">058-01-122</p> <p style="text-align: center;">MDMA 98 mg/tab Caffeine 40 mg/tab</p> | <p style="text-align: center;">Bearded Face</p>  <p style="text-align: right; font-size: small;">055-13-118</p> <p style="text-align: center;">MDMA 88 mg/tab Caffeine 28 mg/tab</p> | <p style="text-align: center;">Butterfly</p>  <p style="text-align: right; font-size: small;">005-01-125</p> <p style="text-align: center;">MDMA 80 mg/tab Caffeine 27 mg/tab</p> |
| <p style="text-align: center;">RN</p>  <p style="text-align: right; font-size: small;">039-05-117</p> <p style="text-align: center;">MDMA 53 mg/tab Caffeine 6 mg/tab</p> | <p style="text-align: center;">Yin-Yang</p>  <p style="text-align: right; font-size: small;">056-01-119</p> <p style="text-align: center;">MDMA 38 mg/tab Caffeine 62 mg/tab</p> | <p style="text-align: center;">(No Logo)</p>  <p style="text-align: right; font-size: small;">099-05-148</p> <p style="text-align: center;">MDMA 100 mg/tab MA 4 mg/tab Ketamine - Trace</p> | <p style="text-align: center;">Crown (Cross)</p>  <p style="text-align: right; font-size: small;">063-09-130</p> <p style="text-align: center;">MDMA 86 mg/tab MA 2 mg/tab Ketamine 5 mg/tab</p> |
| <p style="text-align: center;">SX</p>  <p style="text-align: right; font-size: small;">051-09-134</p> <p style="text-align: center;">MDMA 84 mg/tab MA 3 mg/tab Ketamine 4 mg/tab</p> | <p style="text-align: center;">SX</p>  <p style="text-align: right; font-size: small;">051-10-110</p> <p style="text-align: center;">MDMA 72 mg/tab MA 3 mg/tab Ketamine 4 mg/tab</p> | <p style="text-align: center;">Spider</p>  <p style="text-align: right; font-size: small;">065-06-133</p> <p style="text-align: center;">MDMA 64 mg/tab MA 21 mg/tab Ketamine 43 mg/tab</p> | <p style="text-align: center;">Crown (Ruff)</p>  <p style="text-align: right; font-size: small;">061-01-128</p> <p style="text-align: center;">MDMA 64 mg/tab MA 21 mg/tab Ketamine 43 mg/tab</p> |
| <p style="text-align: center;">V2K</p>  <p style="text-align: right; font-size: small;">070-10-146</p> <p style="text-align: center;">MDMA 43 mg/tab MA 10 mg/tab Ketamine 64 mg/tab</p> | <p style="text-align: center;">Y2K</p>  <p style="text-align: right; font-size: small;">057-05-120</p> <p style="text-align: center;">MDMA 38 mg/tab MA 16 mg/tab Ketamine 48 mg/tab</p> | <p style="text-align: center;">Y2K</p>  <p style="text-align: right; font-size: small;">057-05-121</p> <p style="text-align: center;">MDMA 37 mg/tab MA 16 mg/tab Ketamine 52 mg/tab</p> | <p style="text-align: center;">Mitsubishi</p>  <p style="text-align: right; font-size: small;">011-05-152</p> <p style="text-align: center;">MDMA 5 mg/tab MA 2 mg/tab Ketamine - trace</p> |



| | | | |
|--|--|-------|-------|
| 888  MDA 75 mg/tab | Two Hearts  MDA 54 mg/tab Ephedrine 40 mg/tab | ***** | ***** |
|--|--|-------|-------|

Figure Abbreviations: MA - Methamphetamine; MDA - 3,4-Methylenedioxyamphetamine; MDMA - 3,4-Methylenedioxymethamphetamine.

Table 1. Select Physical and Chemical Characteristics of Ecstasy Tablets Seized in Japan During the First Half of CY-2002.

| ID No. | Logo | Diameter | Thickness (mm) | Weight (mg) | Outside color | Active Ingredients (Percent per Tablet) |
|--------|--------------|----------|----------------|-------------|---------------|---|
| 101 | "B29" | 8.3 | 4.7 | 287 | blue | MDMA (55 %) |
| 102 | "B29" | 8.3 | 4.5 | 285 | brown | MDMA (53 %) |
| 103 | "!" | 8.3 | 3.3 | 205 | yellow | MDMA (31 %) |
| 104 | Bird (Fry) | 7.1 | 3.0 | 140 | blue | MDMA (64 %) |
| 105 | Mickey Mouse | 7.1 | 4.3 | 225 | brown | MDMA (39 %) |
| 106 | Motorola | 8.5 | 3.6 | 140 | brown | MDMA (36 %) |
| 107 | Mitsubishi | 9.3 | 4.4 | 297 | grey | MDMA (22 %) |
| 108 | Tower | 8.2 | 5.0 | 325 | yellow | MDMA (30 %) caffeine (9 %) |
| 109 | Smiley | 7.1 | 3.5 | 168 | orange | MDMA (24 %) |
| 110 | "SX" | 8.1 | 4.3 | 218 | orange | MDMA (33 %) methamphetamine (1 %) ketamine (2 %) |
| 111 | "007" | 7.2 | 4.7 | 224 | brown | MDMA (22 %) |
| 112 | Propeller | 10.1 | 4.4 | 305 | white | MDMA (19 %) |
| 113 | Crocodile | 8.1 | 4.6 | 227 | grey | MDMA (38 %) |
| 114 | Smiley | 7.2 | 4.2 | 202 | green | MDMA (34 %) |
| 115 | Fish | 7.2 | 3.0 | 139 | blue | MDMA (30 %) |
| 116 | "FF" | 7.1 | 5.2 | 256 | yellow | MDMA (31 %) |
| 117 | "RN" | 8.1 | 3.9 | 216 | green | MDMA (25 %) caffeine (3 %) |
| 118 | Bearded Face | 8.2 | 5.2 | 309 | yellow | MDMA (28 %) caffeine (9 %) |
| 119 | Yin-Yang | 8.2 | 4.3 | 259 | white | MDMA (15 %) caffeine (24 %) |
| 120 | "Y2K" | 9.1 | 3.2 | 207 | green | MDMA (18 %) methamphetamine (8 %) ketamine (23 %) |
| 121 | "Y2K" | 9.2 | 3.4 | 209 | green | MDMA (18 %) methamphetamine (8 %) ketamine (25 %) |
| 122 | Popeye | 8.6 | 4.2 | 261 | white | MDMA (38 %) caffeine (15 %) |

| | | | | | | |
|-----|-------------------|-----|-----|-----|--------|---|
| 123 | Two Hearts | 8.1 | 5.1 | 226 | purple | MDA (24 %) ephedrine (18 %) |
| 124 | Mitsubishi | 8.2 | 5.4 | 311 | white | MDMA (20 %) |
| 125 | Armani | 8.9 | 4.7 | 315 | white | MDMA (19 %) |
| 126 | "cK" | 8.2 | 4.9 | 304 | grey | MDMA (32 %) |
| 127 | Hole | 8.5 | 4.5 | 282 | green | MDMA (15 %) |
| 128 | Crown (Ruff) | 8.8 | 5.8 | 204 | white | MDMA (25 %) methamphetamine (5 %) ketamine (10 %) |
| 129 | "XL" | 8.2 | 4.9 | 294 | orange | MDMA (33 %) |
| 130 | Crown (Cross) | 9.1 | 4.1 | 221 | brown | MDMA (39 %) methamphetamine (1 %) ketamine (2 %) |
| 131 | "CU" | 8.1 | 4.6 | 262 | green | MDMA (33 %) |
| 132 | Cellular Phone | 9.2 | 4.3 | 274 | white | MDMA (20 %) |
| 133 | Spider | 9.2 | 7.0 | 260 | blue | MDMA (25 %) methamphetamine (8 %) ketamine (17 %) |
| 134 | "SX" | 8.1 | 4.2 | 234 | brown | MDMA (36 %) methamphetamine (1 %) ketamine (2 %) |
| 135 | Tulip | 8.4 | 6.5 | 348 | brown | MDMA (46 %) |
| 136 | "XL" | 8.2 | 4.8 | 265 | orange | MDMA (55 %) |
| 137 | Star | 8.6 | 6.2 | 328 | green | MDMA (23 %) |
| 138 | Mercedes | 7.1 | 2.6 | 105 | white | MDMA (35 %) |
| 139 | Lozenge | 7.2 | 3.3 | 161 | white | MDMA (35 %) |
| 140 | Superman | 9.2 | 3.6 | 281 | white | MDMA (21 %) |
| 141 | Smurf | 9.1 | 3.8 | 280 | white | MDMA (19 %) |
| 142 | "007" | 8.1 | 5.0 | 311 | red | MDMA (23 %) |
| 143 | no logo | 7.3 | 4.5 | 179 | orange | MDMA (37 %) |
| 144 | Butterfly | 8.0 | 5.0 | 307 | yellow | MDMA (21 %) |
| 145 | Lozenge | 7.1 | 3.3 | 165 | white | MDMA (37 %) |
| 146 | "V2K" | 9.2 | 3.0 | 234 | orange | MDMA (18 %) methamphetamine (4 %) ketamine (27 %) |
| 147 | Mitsubishi | 8.0 | 5.0 | 304 | yellow | MDMA (34 %) |
| 148 | no logo | 8.1 | 4.3 | 254 | green | MDMA (39 %) methamphetamine (2 %) Ketamine trace |
| 149 | "888" | 8.2 | 4.3 | 217 | rose | MDA (35 %) |
| 150 | Mitsubishi | 9.0 | 5.0 | 319 | white | MDMA (20 %) |
| 151 | Butterfly | 9.2 | 4.4 | 326 | green | MDMA (25 %) |
| 152 | Mitsubishi | 9.4 | 5.0 | 306 | green | MDMA (2 %) methamphetamine (1 %) Ketamine trace |
| 153 | Smiley | 7.1 | 3.2 | 180 | orange | MDMA (26 %) |
| 154 | Tower | 8.1 | 5.0 | 333 | yellow | MDMA (32 %) caffeine (11 %) |

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