THE ANALYSIS OF ECSTASY TABLETS BY ICP/MS AND ICP/AES

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ABSTRACT: In this study, tablets coming from different police seizures (in Switzerland) were analysed by ICP/MS and ICP/AES. Two methods of preparation were carried out in order to compare the influence of sample preparation. 25 elements were screened by ICP/AES whereas most of the periodic table could be screened by ICP/MS. Approximative detection limits were determined for most of the elements showing, as expected, a greater sensitivity for ICP/MS. The inter-variability was studied by analysing 20 tablets coming from the same batch and another 20 tablets coming from another batch. The reproducibility of each detection technique was also evaluated. Within day, and depending of the element, the standard deviation can vary between 1 and 10%. The most frequent elements found were Ca, Mg, Na, K, Al, Si and Fe. Full results are presented and discussed.

KEY WORDS: Amphetamines; MDMA; MDEA; Inorganic analyses; Sample comparison.

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INTRODUCTION

Ecstasy tablets have the advantage of providing a wide range of information, namely visual, physical and chemical. This information can be used to link samples and to better understand the illicit manufacture in clandestine laboratories.

Visual and physical characteristics such as the colour, the logo (if present), the dimensions, the shape and the mould defects give valuable information on the manufacture of the tablet itself [4, 5, 6, 23].

Chemical analyses which include the identification of the illicit drug and of the adulterants and/or diluents provide as well important information on the variety of tablet contents [2].

As for the illicit drug itself, it often contains impurities such as, reaction by-products, intermediates and precursors. The analyses of these impurities which provide a chemical profile have been widely explored for amphet-

amine and metamphetamine and have been described by many authors such as [7, 8, 11, 14, 20, 21, 24] and many others.

Classification and automatic comparison methods using these chemical profiles have also been developed for these compounds [9, 10, 17].

Regarding other amphetamine derivatives such as MDA, MDMA and MDEA, some publications have also studied the corresponding impurities but they are much more seldom [2, 3, 15, 18, 25, 26, 27, 28].

In relation to inorganic analyses, only a few publications have studied the elemental content of amphetamine-type drugs. These were limited to metamphetamine powder [3, 16, 22].

This preliminary study on inorganic analyses of Ecstasy tablets had multiple aims. The first one was to compare two different methods available to us in order to assess the most suitable one, namely ICP/MS (Inductively Coupled Plasma Mass Spectrometry) and ICP/AES (Atomic Emission Spectrometry).

The second aim was to compare two different sample preparation methods:

1. direct dissolution in dilute nitric acid, and
2. the same method followed by microwave digestion.

Then, with the samples available, we wanted to determine the variation within batch and do a screening study of street samples.

The results were also expected to give answers to some questions such as:

- What elements are detected in ecstasy tablets? How many? In which concentration?
- Is it possible to link samples by their elemental "profile"?
- Can we detect traces of catalysts and/or metals coming from chemicals used for the synthesis of these amphetamines?

INSTRUMENTATION

ICP/AES

The analyses by ICP/AES were carried out on a Perkin Elmer Emission Spectrometer Plasma 1000, thanks to the department of analytical chemistry of the University of Lausanne.

The following elements were analysed: P, Zn, Pt, Pb, Ni, Fe, Co, Au, B, Si, Hg, Mn, Cr, Mg, V, Cu, Ag, Ti, Pd, Ca, Al, Sr, Ba, Na, Li and K.

The photomultiplicator voltage was 600 V except for Ca (400 V), Mg (400 V), Hg (800 V), Pt (800 V), Pb (800 V), Pd (800 V) and K (800 V).

The wavelengths used were the following: P (213.618 nm), Zn (213.856 nm), Pt (214.423 nm), Pb (220.353 nm), Ni (231.604 nm), Fe (238.204 nm),

Co (238.892 nm), Au (242.795 nm), B (249.773 nm), Si (251.611 nm), Hg (253.652 nm), Mn (257.610 nm), Cr (267.716 nm), Mg (279.553 nm), V (292.402 nm), Cu (324.754 nm), Ag (328.668 nm), Ti (334.941 nm), Pd (340.458 nm), Ca (393.366 nm), Al (396.152 nm), Sr (407.771 nm), Ba (455.403 nm), Na (589.592 nm), Li (670.781 nm) and K (766.490 nm).

For quantification, calibration curves were calculated by measuring blanks and standard solutions with a concentration range between 0.025 and 20 ppm depending on the elements responses. The exceptions were Au, Ag, Pd and Li where standards solutions were not available. Phosphorous was measured but was then discarded.

ICP/MS

The analyses by ICP/MS were performed on a Perkin Elmer Elan 6000 kindly made available by the Army Laboratory in Spiez, Switzerland.

73 elements of the periodic table were analysed. These were quantified by measuring blanks and standard solutions of Li (100 ppb), Be (1000 ppb), B (1000 ppb), Na (100 ppb), Mg (100 ppb), Al (100 ppb), K (100 ppb), Ca (1000 ppb), V (100 ppb), Cr (100 ppb), Mn (100 ppb), Fe (1000 ppb), Co (100 ppb), Ni (100 ppb), Cu (100 ppb), Zn (1000 ppb), Ga(100 ppb), As (1000 ppb), Se (1000 ppb), Rb (100 ppb), Sr (100 ppb), Mo (100 ppb), Ag (100 ppb), Cd (100 ppb), Te (100 ppb), Ba (100 ppb), Tl (100 ppb), Pb (100 ppb) and Bi (100 ppb).

The other elements were calibrated by interpolation. The quantification was carried out using the Totalquant® method.

SAMPLE PREPARATION

a) Microwave: The tablet is crushed. 200 mg are introduced into a teflon cylinder. 5 ml of concentrated HNO_3 and 1 ml of H_2O_2 are added. The cylinder is closed and put on a turning plate in an adapted microwave oven (6 cylinders in total). A special oven programme (50 min) was used. The cylinders were let to cool down for 45 min. The extracts were transferred to polypropylene tubes and the volume adjusted to 25 ml with pure HNO_3 2%.

b) Dilute HNO_3: 25 ml of dilute HNO_3 (5%) are added to 200 mg of crushed tablet in a 50 ml polypropylene tube. The tube was vortexed and put in an ultrasonic bath for 10 min. It was centrifuged and the supernatant was filtered into another tube through a PTFE disk filter.

ELEMENTS POTENTIALLY PRESENT IN TABLETS

Before starting the analyses, it is useful to draw a list of elements that are expected to be found in ecstasy tablets. The source can come from various compounds such as diluents, lubricants or dyes that are involved in the manufacture of the tablet itself [6, 19]. Regarding the illicit drug, metals contained in chemicals involved in the synthesis process might also be found [1, 3].

1. Diluents: Na, Ca, Ba (sulphate, phosphate salts).
2. Lubricants: Mg, Zn, Al, Li (stearates) and B (boric acid).
3. Catalysts: Pd, Pt, Ni, etc.
4. Reduction of intermediate ketones: $LiAlH_4$, $NaBH_3CN$, $Al/HgCl_2$.
5. Preparation of intermediate ketones: Fe/HCl, $SnCl_2$, Pb $(OAc)_2$, $PdCl_2$/CuCl, MeZnI, etc.
6. Various: TiO_2 (E 171).
7. Dyes: I (E 127), Fe (E 172), Cu (E 141)

RESULTS

Comparison between ICP/AES and ICP/MS, detection limits and relative standard deviations

The following table illustrates the different detection limits obtained with ICP/AES and ICP/MS. As can be seen, ICP/MS is much more sensitive but differences vary depending on the element analysed.

TABLE I. DETECTION LIMITS FOR ICP/MS AND ICP/AES

Elements	ICP-AES (in ppm)	ICP-MS (in ppm)	Ratio
Ca	0.05	0.0346	1.4
Ba	0.005	0.003	1.7
Al	0.1	0.0381	2.6
Zn	0.1	0.0289	3.5
Mg	0.1	0.0252	4.0
Fe	0.1	0.0136	7.4
Na	0.5	0.0375	13.3
Sr	0.005	0.0003	16.7
Ti	0.1	0.005	20.0
Si	0.1	0.0038	26.3
Cu	0.05	0.0016	31.3
B	0.1	0.0014	71.4
V	0.1	0.0014	71.4
K	2	0.0256	78.1
Mn	0.05	0.0004	125.0
Ni	0.2	0.0013	153.8
Cr	0.2	0.0008	250.0
Pb	0.2	0.0005	400.0
Co	0.2	0.0003	666.7
Pt	0.2	0.0002	1000.0

The relative standard deviations for ICP/AES can vary between 1.5 and 5% depending on the element analysed (5 measurements of the same sample within one day).

For ICP/MS, it varies between 1 and 10% depending on the element analysed (10 measurements of the same sample within one day).

Comparison of sample preparation methods A and B

In order to compare the two sample preparation methods, 28 tablets coming from one batch (containing MDEA) were analysed by ICP/MS. Results are illustrated below in Figures 1 and 2.

Regarding the elements detected, it can be seen that the microwave method gives much better results for Mg, Na, Si, Pt, Al and Fe.

For Ca, K and even Ti, no significant differences were observed.

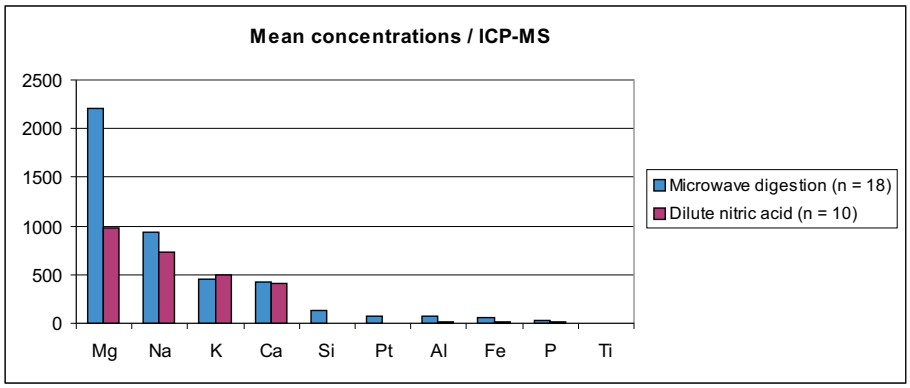

Fig. 1. Mean concentrations in ppm.

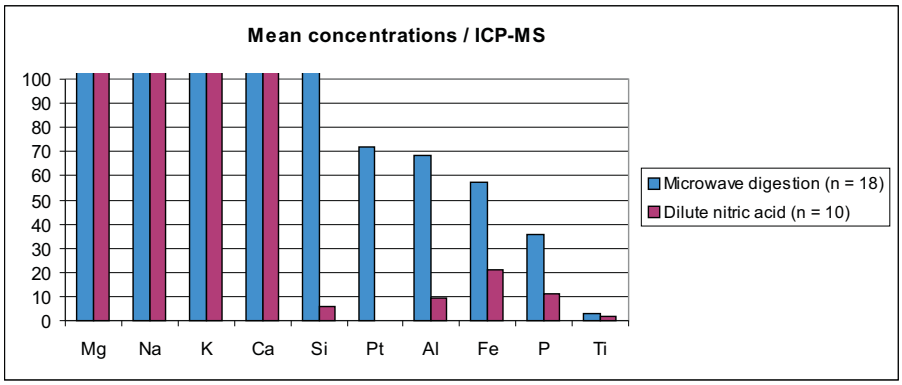

Fig. 2. Expanded view of Figure 1.

Variation within batch

20 tablets coming from one batch (containing MDMA) and 20 other tablets coming from another batch (containing MDEA) were used to study the variation within batch. The same prepared samples were then also analysed by ICP/AES. Results are shown in Table II.

TABLE II. RELATIVE STANDARD DEVIATIONS FOR ICP/MS AND ICP/AES

Elements	ICP/AES	ICP/MS
Fe	34.5	45.1
Si	27.5	37.5
Mg	17.3	22.4
Ca	10.1	16.9
Al	18.4	23.8
Na	13.0	16.9
K	9.8	21.5
B	20.3	20.4
Cu	19.4	18.6
Pt	28.9	33.6

The variation depends on the element analysed. The mean variation was found to be 20% for ICP/AES and 25% for ICP/MS. Therefore, these relative standard deviations represents the variation within batch including the instrument and quantification errors.

It has to be emphasised that the relative standard deviations are independent of the preparation method. Similar values were found between the microwave method and the direct dissolution in dilute nitric acid.

Screening study of street samples

43 tablets from 43 different seizures were analysed by ICP/MS (prepared by the microwave method). 49% of the tablets contained MDMA, 33% MDEA, 14% amphetamine, 2% MBDB and 2% MDA.

Moreover, 4 other samples which did not contain any illicit drugs were analysed. They consisted of lactose powder, one tablet of Triludan® (antihistaminic), one tablet containing a mixture of cafeine, ephedrine, testosterone, sucrose and traces of yohimbine (tablet 109) and another tablet containing a mixture of cafeine, paracetamol, ephedrine, quinine and lactose (tablet 26).

With ICP/MS, a high background was observed meaning that almost every element was detected at very low concentrations (ppb level). Therefore, only the values above 1 ppm were taken into account.

Table III shows the results obtained.

TABLE III. PERCENTAGE OF ELEMENTS AND RANGE OF CONCENTRATIONS FOUND IN ECSTASY TABLETS

Elements	% found in tablets	Range (in ppm)	Elements	% found in tablets	Range (in ppm)
Zn	30.2	3–53	Mg	100.0	86–4538
Pt	41.9	2–73	V	2.3	1.4
Pb	11.6	1.7–4	Cu	46.5	1–19
Ni	18.6	1 -25	Ti	51.2	1.5–265
Fe	69.8	2–760	Ca	100.0	8–151876
Co	2.3	1.6	Al	67.4	10–1018
B	44.2	1–3782	Sr	4.7	1.4–32
Si	74.4	1–296	Na	100.0	8–14986
Hg	4.7	1–2.6	K	81.4	6–497
Mn	20.9	1.2–6	I	25.6	1.6–325
Cr	44.2	1–125	Sn	7.0	1.3–2

It can be seen that every tablet contained Ca, Na and Mg in high concentrations. This is very probably due to sulphate and phosphate salts that are commonly used in the manufacture of tablets. For Mg, the stearate salt is very common as lubricant.

A high percentage of tablets contained Fe, B, Si, Cr, Cu, Ti, Al and K. Still, these elements were also detected in the four other samples that do not contain illicit drugs (see Table IV below). Therefore, the origin of these elements are more difficult to interpret.

As for some other elements, their presence will be discussed later.

TABLE IV. ELEMENTS FOUND IN 4 OTHER SAMPLES CONTAINING NO ILLICIT DRUGS (IN PPM)

Other	Fe	B	Si	Cr	Mg	Cu	Ti	Ca	Al	Na	K	I	Sn
Triludan®		1.4		1.3	453	2.1		36		31169	36	1.4	
Lactose		1.5	13		3.3			12		21	80		
109	9	4		1.2	4049	1.8		454		42091			
26	61		19		2336	2	2	434	29	17318	1053		4.7

Examples

Finally, some examples are presented below to illustrate our results.

Example 1

Four tablets (green with trefoil logo) which have the same visual, physical and chemical characteristics (MDMA HCl approx. 40% and lactose) were analysed. The elements detected are shown in Figure 3.

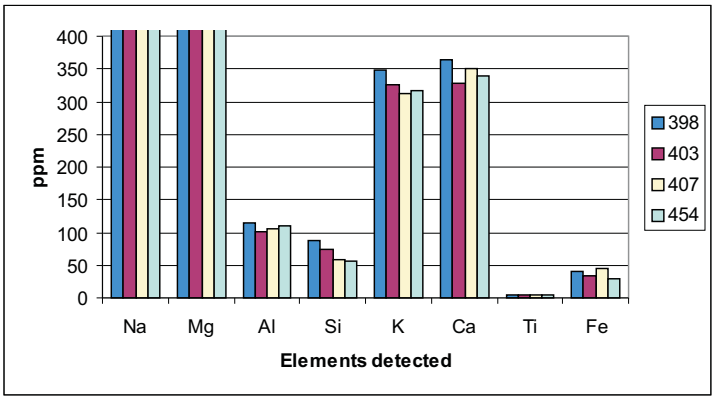

Fig. 3. Elements detected in four similar MDMA tablets.

The values for Na and Mg are intentionally out of scale in Figure 3. Measured values for Na were between 2200 and 2500 ppm. For Mg, it varied between 1700 and 2100 ppm.

Although variations were observed (similar to those described in section 4.3), the inorganic profile is quite similar. Of course, the more elements are detected, the easier is the interpretation.

Example 2

In this case, five tablets (white with the Twins logo) with the same visual, physical and chemical characteristics (MDMA HCl approx. 40% and sorbitol) were analysed. This time, slight differences were observed in the colour of the tablets (different kinds of white). Results are shown in Figure 4.

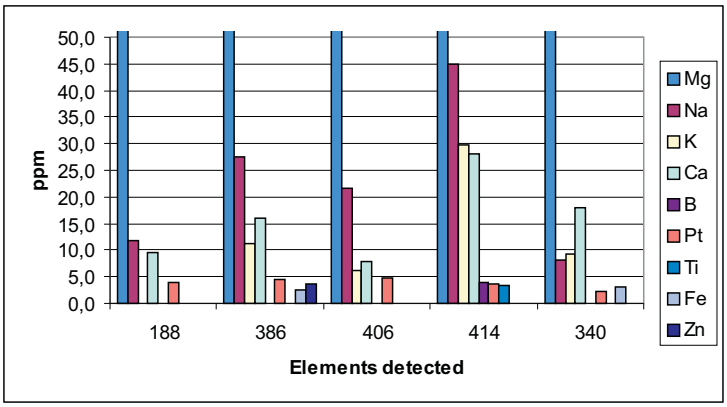

Fig. 4. Elements detected in five similar MDMA tablets.

In this case, the five tablets have different inorganic profiles:

1. There are significant variations in the concentrations of Ca and Na (and amounts of Mg vary between 1900 and 2800 ppm).
2. The tablet 188 doesn't contain any K and the concentrations vary in the other four tablets.
3. The tablet 414 is the only tablet containing B and Ti.
4. The tablets 340 and 386 are the only ones containing Fe.
5. The tablet 386 is the only one containing Zn.

Still, all tablets contain a small amount of Pt (between 2 and 5 ppm).

Example 3

An additional analysis was performed on a sample of amphetamine sulphate which was synthesised in the laboratory. Phenyl-2-propanone was reduced with ammonium acetate and sodium cyanoborohydride. The aim was to see if boron (B) would be detected. The sample was analysed by ICP/MS and prepared via the microwave method. Results are shown in Table V.

TABLE V. ELEMENTS DETECTED IN A HOME-MADE AMPHETAMINE SULPHATE SAMPLE

Elements	In ppm
Na	14.4
Mg	3.6
Si	417
Ca	16.6
Cr	1.8
Cu	1.2
Ag	20.6

The high concentration of silver (Ag) was found to be a result of vessel contamination. Indeed, the test tube used for crystallisation of the sulphate salt was contaminated with silver nitrate. It was used for an anion test (chloride) and not washed properly.

Example 4

In this case, two tablets having completely different visual and physical characteristics were found to have a very similar inorganic profile (see Figure 5). Both tablets contained MDEA HCl (around 40%) and lactose.

Tablet 88 has the "Sonic" logo, weighs 265 mg, with a diameter of 8.5 mm and a width of 4.1 mm.

Tablet 47 has the "Adam" logo, weighs 235 mg, with a diameter of 9 mm and a width of 3.1 mm.

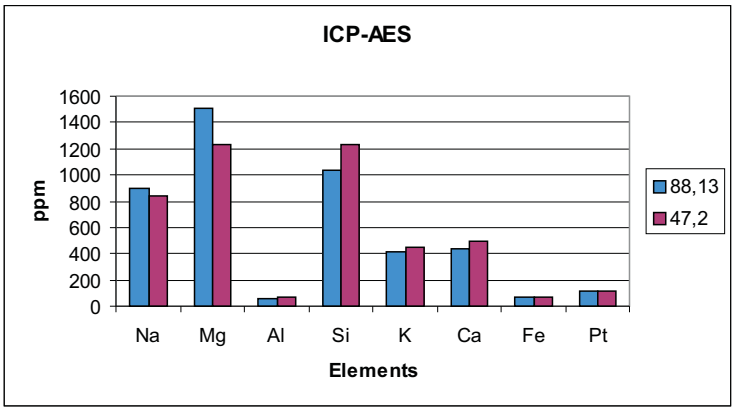

Fig. 5. Elements detected by ICP-AES in two tablets with different visual and physical characteristics.

In this case, an analysis by GC/MS was carried out to detect the organic impurities in these two tablets. The result is illustrated in Figure 6.

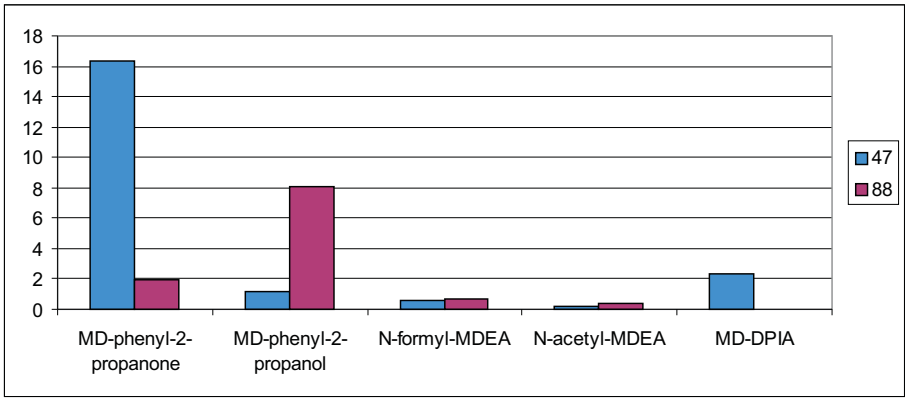

Fig. 6. Proportions of organic impurities in samples 47 and 88.

The organic analysis by GC/MS, which was similar to the one used for amphetamine profiling, revealed the presence of five impurities, namely: 3,4-(methylenedioxy)phenyl-2-propanone, 3,4-(methylenedioxy)phenyl-2-propanol, N-formyl-MDEA, N-acetyl-MDEA and Di-[1-(3,4-methylenedioxy)phenyl-2-propyl]amine.

The ratio of the two first impurities are totally different and the fifth impurity was only present in the tablet 47 indicating that the MDEA contained in these tablets is very probably not coming from the same batch.

Moreover, a similar analysis was performed to detect the fatty acids and their relative concentration in the two tablets (results not shown here). It was determined that the tablet 88 contained three times more fatty acids than tablet 47 indicating that the tablets were probably not manufactured in the same way.

DISCUSSION

Detection limits

As can be seen in Table I, ICP/MS is much more sensitive than ICP/AES but the differences vary depending on the element analysed.

Relative standard deviations

Table II show that the relative standard deviations are quite high with the quantitation methods used. The variations depend on the elements analysed. The total mean variation including instrumentation error, quantitation error and inter-variation within batch is between 20 and 25%. The methods used should therefore be considered as semi-quantitative. Nevertheless, if a high number of elements are detected, a classification is still possible. Moreover, with the exception of example 4 (see section "Examples"), all unrelated samples were discriminated by their elemental profiles.

Sample preparation

The microwave method is more effective but requires special equipment. Still, for some elements, no significant differences were observed.

Regarding the preparation time, the microwave method is much more time-consuming and less practical.

Screening study

This showed interesting results but also showed that elements found in low concentrations and only in a few tablets are difficult to interpret.

For example, Pb, Ni, Co, Hg, Mn, V, Sr and Sn were found in low concentrations and in a minority of tablets. Although some of these elements can be found in chemicals used for the synthesis of illicit amphetamine derivatives (see section "Elements potentially present in tablets"), it is more probable that they originate from external contamination or represent some kind of background noise.

More often found were Zn, Fe, B, Cr, Cu, Ti, Al and I. The presence of Zn, B, Al and Ti could be explained by the fact that they are used in the manufacture of tablets. Zinc and aluminium stearates as well as boric acid are lubri-

cants and titanium oxide is used as a "whitener". This is especially true when high concentrations are found. For example, two tablets contained respectively 816 and 3782 ppm of B, 14 tablets contained over a 100 ppm of Al and 4 tablets contained over a 100 ppm of Ti.

As for Fe, it's a common element. It was also found in tablets containing no illicit drugs (up to 60 ppm). Still, the concentrations vary quite a lot and 3 tablets were found to contain over 200 ppm of Fe. In that case, external contamination or background noise seems unlikely. Fe powder is used in the manufacture of the intermediate ketone 3,4-(methylenedioxy)phenyl-2-propanone. Therefore, this could be one possible explanation but still has to be demonstrated.

44% of tablets contained Cr but the concentration did not exceed 9 ppm (in general, the values were close to 1 ppm) except in one case where 125 ppm were found. Again, external contamination or background noise looks unlikely in this latter case.

46% of tablets contained Cu but the highest value did not exceed 19 ppm. Therefore, its presence is difficult to interpret as it was also found in a pharmaceutical product (Triludan®) and in two other tablets which did not contain any illicit drugs.

Iodine is interesting. In some cases, it's presence could be explained. A pink dye named erythrosine (E 127) contains 4 atoms of I in its chemical structure. It was identified in two pills. These two pills were found to contain respectively 88 and 325 ppm of I.

Potassium (K) was found in 80% of the samples but again, its presence is difficult to interpret as it was found in the Triludan® tablet (36 ppm), in a tablet containing no illicit drugs (1053 ppm) and in lactose (80 ppm). The same type of comment can also be applied to Si (found in 75% of samples).

Finally, platinum (Pt) has interesting features. It was found in 42% of the tablets (between 2 and 73 ppm).

It is known that platinum oxide is quite common as a catalyst in the manufacture of amphetamine-type of drugs. It has the advantage that it can be re-used many times. Therefore, this could be the reason for its presence in these samples giving an indication of the synthetic route. But again, this has to be demonstrated.

Examples

Example 1 shows 4 tablets which have the same visual, physical, chemical characteristics and a very similar inorganic profile. This is probably a confirmation that these tablets have the same history and originate from the same batch.

On the other hand, example 2 shows the opposite. Although slight visual variations were observed in these five tablets (different tints of white), the

physical and chemical characteristics were the same. But the five tablets could be differentiated by their elemental profiles. In this case, the tablets probably don't have the same history or were not manufactured at the same time. An organic profile would have helped in the interpretation of these results. Unfortunately, this was not possible in this case.

Synthesised amphetamine was analysed in example 3. Although sodium cyanoborohydride was used in the synthesis, no boron (B) was detected. Still, this example shows that contamination of the vessel (Ag) can be detected by ICP. Therefore, this is another factor that has to be taken into account. Presence of elements in a inorganic analysis can be due to vessel contamination.

Mg, Si, Ca, Cr and Cu were also detected in this sample although no chemicals containing these elements were used. This is, therefore, very probably some kind of background noise. This also has to be taken into consideration in the interpretation of results.

In example 4, a very similar elemental profile was observed between two tablets having completely different visual and physical characteristics. Both contained MDEA HCl (about 40%) and lactose. This could be a good example of a false positive link between two samples if inorganic analysis is taken alone. Indeed, the organic profile as well as the fatty acids content are completely different showing that the illicit substance was not manufactured in the same way or at the same time and that the tablets themselves are not coming from the same batch.

Therefore, elemental analysis alone is probably not enough to link samples.

CONCLUSIONS

First, the main aim of this study was to perform a preliminary screening of ecstasy tablets with an elemental analysis technique. The idea was to determine what elements are present and in which range of concentration. The interest was also focused on the variation between same and different batches.

In terms of methods, this study showed that ICP/MS is more sensitive than ICP/AES for inorganic analysis of ecstasy tablets. The microwave method for sample preparation is generally more efficient than simple dissolution in dilute nitric acid but special equipment such as an adapted microwave system is required and the method is more time-consuming.

The methods used in this study are semi-quantitative. Relative standard deviations between 20 and 25% were found for both ICP techniques which includes instrumentation error, quantitation error and inter-variation

within batch. Nevertheless, when sufficient elements are detected, it is possible to establish an elemental profile and differentiate samples.

However, it is believed that the quantitation method can be improved, especially for ICP/MS, thus reducing the relative standard deviations, but this was not the aim of the study.

Moreover, an example showed that the inorganic profile alone is not enough to establish a link between samples. Additional information such as the visual, physical and chemical characteristics (including the organic profile) are necessary in order to correctly interpret the results.

This study also showed that many elements are detected at the ppm level. The interpretation of results is not easy as the presence of these elements is often difficult to explain.

Traces of catalysts and/or metals coming from chemicals used for the synthesis of these illicit amphetamines are not always detected. An example showed that boron (B) was not detected although sodium cyanoborohydride ($NaBH_3CN$) was used in the synthesis. On the other hand, many samples contained platinum (Pt) which could indicate that platinum oxide was used as a catalyst in the illicit synthesis.

Traces of elements due to external contamination can also be detected as it has been shown in the vessel contamination example (see example 3).

Finally, it may prove useful to carry out controlled syntheses via various common routes. The inorganic analyses of the final products would then determine if elements coming from the chemicals used in the synthesis are detected or not and in which concentration. This would probably help in the future interpretation of results.

References:

1. Allen A., Cantrell T. S., Synthetic reductions in clandestine, amphetamine and metamphetamine laboratories: a review, *Forensic Science International* 1989, vol. 42, pp. 183–199.
2. Bohn M., Bohn G., Blaschke G., Synthesis markers in illegally manufactured 3,4-methylenedioxyamphetamine and 3,4-methylenedioxymethylamphetamine, *International Journal of Legal Medicine* 1993, vol. 106, pp. 19–23.
3. Dal Cason T. A., An evaluation of the potential for clandestine manufacture of 3,4-methylenedioxyamphetamine (MDA) analogs and homologs, *Journal of Forensic Sciences* 1990, vol. 35, pp. 675–697.
4. Gomm P. J., A technique for the physical examination of illicitly made tablets, *Journal of Forensic Science Society* 1975, vol. 15, pp. 219–226.
5. Gomm P. J., Hugues R. J., Examination of illicity produced tablets and capsules, [in:] The analysis of drugs of abuse, Gough T. [ed.], John Wiley & Sons, Chichester 1991.

6. Gomm P. J., Humphreys I. J., Identification of the major excipients in illicit tablets using infrared spectroscopy, *Journal of Forensic Science Society* 1975, vol. 15, pp. 293–299.
7. Huizer H., Theeuwen A. B. E., Verweij A. M. A. [et al.], Impurities in illicit amphetamine, *Journal of Forensic Sciences Society* 1981, vol. 21, pp. 225–234.
8. Inoue T., Tanaka K., Ohmori T. [et al.], Impurity profiling analysis of methamphetamine seized in Japan, *Forensic Science International* 1994, vol. 69, pp. 97–102.
9. Jonson S., Strömberg L., Two-level classification of Leuckart amphetamine, *Forensic Science International* 1994, vol. 69, pp. 31–44.
10. Kärkkäinen M., Sippola E., Pikkarainen A. L. [et al.], Automated gas chromatographic amphetamine profiling, *Forensic Science International* 1994, vol. 69, pp. 55–64.
11. King L. A., Clarke K., Orpet A. J., Amphetamine profiling in the UK, *Forensic Science International* 1994, vol. 69, pp. 65–75.
12. King L. A., Drug content of powder and other illicit preparations in the UK, *Forensic Science International* 1997, vol. 85, pp. 135–147.
13. Kishi T., Analysis of trace elements in methamphetamine hydrochloride by inductively coupled plasma-mass spectrometry, *Journal of Research of National Bureau of Standards* 1988, vol. 93, pp. 469–471.
14. Kram T. C., Kruegel A. V., The identification of impurities in illicit metamphetamine exhibits gas chromatography/mass spectrometry and nuclear magnetic resonance spectrometry, *Journal of Forensic Sciences* 1977, vol. 22, pp. 40–52.
15. Lukaszewski T., Spectroscopic and chromatographic identification of precursors, intermediates, and impurities of 3,4-methylenedioxyamphetamine synthesis, *Journal of the Association of Official Analytical Chemists* 1978, vol. 61, pp. 951–967.
16. Marumo Y., Inoue T., Seta S., Analysis of inorganic impurities in seized metamphetamine samples, *Forensic Science International* 1994, vol. 69, pp. 89–95.
17. Perkal M., Pearson J. R., Impurity profiling of methlyamphetamine in Australia and the development of a national drugs database, *Forensic Science International* 1994, vol. 69, pp. 77–87.
18. Renton R. J., Cowie J. S., Oon M. C. H., A study of the precursors, intermediates and reaction by-products in the synthesis of 3,4-methylenedioxymethylamphetamine and its application to forensic drug analysis, *Forensic Science International* 1993, vol. 60, pp. 189–202.
19. Rhodes E. F., Thornton J., Occurrence of excipient materials in illicit tablet manufacture, *Microgram* 1979, vol. 12, pp. 98–106.
20. Strömberg L., Bergkuist H., Edringsghe E. A. M. K., Comparative gas chromatographic analysis of narcotics. IV metamphetamine hydrochloride, *Journal of Chromatography* 1983, vol. 258, pp. 65–72.
21. Strömberg L., Maehly A. C., Comparative gas chromatographic analysis of narcotics, II amphetamine sulphate, *Journal of Chromatography* 1975, vol. 106, pp. 335–342.

22. Suzuki S. I., Nagao T., Analyses of impurities in metamphetamine by inductively coupled plasma mass spectrometry and ion chromatography, *Journal of Chromatography* 1988, vol. 437, pp. 322–327.
23. Tillson A. H., Johnson D. W., Identification of drug tablet and capsule evidence as to source, *Journal of Forensic Sciences* 1974, vol. 19, pp. 873–883.
24. Verweij A., Impurities in illicit drug preparations: amphetamine and metamphetamine, *Forensic Science Review* 1989, vol. 1, pp. 1–11.
25. Verweij A., Clandestine manufacture of 3,4-methylenedioxymethamphetamine (MDMA) by low-pressure reductive amination. A mass spectrometric study of some reaction mixtures, *Forensic Science International* 1990, vol. 45, pp. 91–96.
26. Verweij A., Impurities in illicit drug preparations: 3-,4-(methylenedioxy)amphetamine and 3,4-(methylenedioxy)metamphetamine, *Forensic Science Review* 1989, vol. 4, pp. 138–145.
27. Verweij A., Verunreinigungen in illegalem Amphetamin, basische Verunreinigungen in auf dem Drogenmarkt käuflichem 3,4-(Methylendioxy)methylamphetamin, *Archiv für Kriminologie* 1991, Bd 188, S. 154–158.
28. Verweij A., Sprong A., Verunreinigungen in illegalem Amphetamin, verunreinigungen, die bei der Herstellung von 3,4-(Methylendioxy)amphetamin (MDA) durch Kondensation zwischen Nitroethan und piperonal aufgefunden werden, *Archiv für Kriminologie* 1992, Bd 190, S. 24–28.