B60 XTC Characterisation Using ICPMS

Gerard J.Q. van der Peijl, PhD, Claudia P.H. van den Boom, Ing, Annabel Bolck, PhD, and Andrew M. Dobney, PhD, Netherlands Forensic Institute of the Netherlands Ministry of Justice, PO Box 3110, Rijswijk, 2280 GC, Netherlands*

After attending this presentation, attendees will the participant will appreciate the possibilities and limitations of ICPMS for XTC comparison investigations.

This presentation will demonstrate new interesting forensic applications of ICPMS elemental and isotopic techniques which are presently developed and promise to result in much more strongly discriminating methods for forensic applications. The subject in the present presentation is just one of these applications but is very useful in demonstrating the relevance of these techniques. The relevance is also recognized by the international NITECRIME forensic network that has become active in this field. More information on this network can be submitted if requested.

Introduction. XTC (3,4-methylenedioxymethamphetamine or MDMA) is presently one of the most favored illegal party drugs. Important illegal production facilities are situated in the Netherlands. At the NFI, new analytical chemical methods are being developed to characterize and compare XTC materials. Results will be presented on the development and application of an ICPMS (Inductively Coupled Plasma Mass Spectrometry) method for this purpose. It is expected that such methods can be used to assist in discrimination between XTC materials from different producers and perhaps even between different production batches. Results from this technique will be combined with results from other techniques such as GC-MS and XRF to maximize discrimination.

Project goal. The goal of this project was to develop a validated forensic method and not so much to build a comprehensive collection of XTC data. However, as part of the validation process about 100 different XTC materials were investigated to obtain an indication of the variation in composition and the discrimination power of the method. Almost 100 apparently unrelated XTC samples from different seizures were supplied.

Method development. Early on in the project, it was decided to focus method development on sample digestion and ICPMS analysis of the resulting solutions. For method development and validation purposes, a large supply of homogeneous XTC powder was prepared by crushing and pooling a number of XTC tablets from one seizure. These tablets were selected on the basis of availability and their anticipated homogeneity.

Individual XTC tablets were crushed inside polythene bags, rather than being milled, to prevent contamination from metals present in milling equipment. The resulting powder was homogenized by coning and quartering. 200 mg of the resulting material was placed inside precleaned quartz microwave digestion vessels into which concentrated nitric acid (5 ml, 65% *m/m*) and hydrogen peroxide (1 ml, 30% *v/v*) were then pippetted. These are typical ICPMS digestion reagents suitable for many sample types and bring the elements present in the samples into solution. The nitric acid dissolves the inorganic components whilst the hydrogen peroxide oxidizes any organic material.

Sample digestion. Samples were digested in a closed vessel microwave digestion unit (Multiwave, Anton Paar, Austria). The digestion scheme was based on a published scheme used by Comment *et al*. but simplified since in our system we have pressure and temperature regulation feedback. Step 1 was a linear ramp from 700 watt to 1000 W over 15 min., step 2 was 1000 W for 10 min. and step 3 a cooling down period of 30 min. During step 2 the irradiation power is controlled by a feedback loop such that a pressure of 75 bar was maintained. Under these conditions, the temperature during step 2 was typically 280°C. The digestion was optimized by varying the length and irradiation power of steps 1 and 2. The concentrations of selected elements (Mg, Al, Ca, Mn, Fe, Zn, Sr, Ba) were determined by ICP AES to arrive at the final digestion conditions. After cooling, the samples were transferred to 50 mL plastic vessels (Sarstedt tubes) and diluted gravimetrically to volume with Milli-Q water. Just prior to analysis, an aliquot of each sample was diluted 10x to a final acid concentration of 3 % m/m HNO3. An internal standard mixture (Sc, Rh, Re) was added to these final dilutions.

Literature on the inorganic composition of illicit drugs is relatively small and even more limited for XTC. Two particularly invaluable sources are Comment et al. [1] and Goldmann (thesis ref) [2]. From these and other sources, the following initial list of elements, potentially present in XTC, was derived (with isotopes to be measured): ^{23}Na, ^{24,25}Mg, ^{43,44}Ca (likely present at high concentrations); ^{27}Al, ^{29,30}Si, ^{39,41}K, ^{47}Ti, ^{52,53}Cr, ^{57}Fe, ^{63,65}Cu: (v possibly present at high concentrations); ^{7}Li, ^{11}B, ^{55}Mn, ^{58,60}Ni, ^{64,66}Zn, ^{81}Br, ^{85}Rb, ^{88}Sr, ^{96,98}Mo ^{106,108}Pd, ^{121,123}Sb ^{132}Cs, ^{137,138}Ba, ^{194,195}Pt, ^{197}Au ^{202}Hg, $^{203, 205}$Tl, ^{208}Pb: (likely present at low concentrations). The anticipated concentrations are based largely on work carried out at the University of Lausanne. The presence of these elements can be attributed to catalyst residues, excipients, colourants etc. This list is not as extensive as for the multi-elemental analysis of heroin or cannabis where the soil composition influences the elements found in those drugs. The synthetic nature of XTC can be expected to preclude the kind of variation (especially rare earth elements) found in natural drugs.

ICPMS conditions. All ICPMS experiments used a quadrupole instrument (6100 DRC, Perkin-Elmer). Relevant instrumental parameters were: 1250 W rf forward power, 1.15 L/min nebulizer gas flow,

glass concentric spray chamber with a Meinhard nebulizer (sample uptake rate ~ 1 or 0.4 mL/min

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depending on conditions). Daily performance and calibration experiments were performed to verify adequate instrumental performance. The ICPMS method was developed and refined in stages, the most important of which are described; e.g, some of the elements in the above list were not found in any of the samples so that a modified list was used. The influence of various other experimental parameters were investigated to result in a reliable analysis method. The absence of matrix effects was demonstrated by standard addition calibrations yielding the same gradients as external calibrations. Quantification was by external calibration with internal standardization.

Results. Preliminary results of the statistical analysis of the ICPMS data showing possible relations between the samples are presented. References. [1] Analyse élémentaire de pilules d'ecstasy par ICP-AES et ICPMS, Stéfane Comment, Université de Lausanne, mai 1998. [2] These de doctorat "l'Analyse des colorants presents dans les comprimes illicites," Till Goldmann, Licencie en Sciences forensiques de l'Université de Lausanne, 2000.

XTC, ICPMS, ICP AES

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