TITLE
Microcrystalline identification of selected designer drugs

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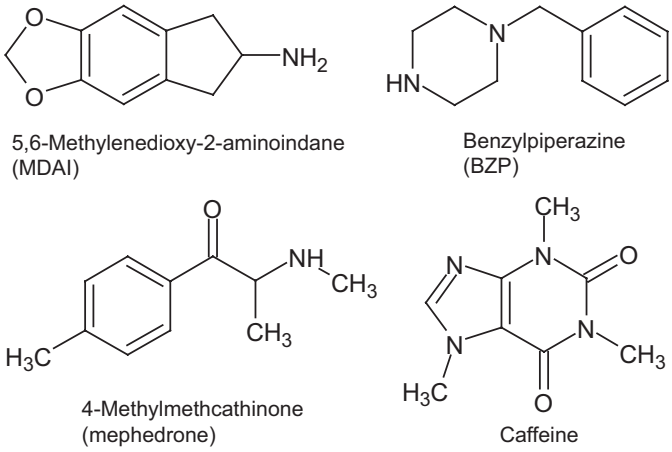
ABSTRACT
A microcrystalline test for the detection of 4-methylmethcathinone (mephedrone), benzylpiperazine (BZP) and 5,6-methylenedioxy-2-aminoindane (MDAI) using aqueous solutions of mercury chloride is described. Each of the compounds investigated formed specific drug-reagent crystals within minutes. The uniqueness of the test was confirmed by comparison of the microcrystalline response to that of other psychoactive stimulants and a common cutting agent. The limit of detection and cut-off levels for reference standards were established to 3 g/L and 5 g/L for mephedrone, 0.5 g/L for MDAI and 0.2 g/L and 0.3 g/L for BZP, respectively. Various mixtures of standards of either mephedrone, BZP or MDAI combined with caffeine were investigated for their microcrystalline response. Results showed that simultaneous detection of drug and cutting agent was possible with the concentrations tested but were dependant on the ratio of drug to cutting agent. BZP could be detected alongside caffeine from as low as 20 % (v/v), MDAI from 40 % (v/v) and mephedrone from 50 % (v/v) and higher. Finally, seven samples of online purchased 'legal highs' were analysed using the developed test and the findings were compared to FTIR and GC-MS results. It was shown that 6 out of 7 samples did not contain the advertised active ingredient. Five samples consisted of BZP, caffeine and 1-[3-(Trifluoromethyl)phenyl]piperazine (3-TFMPP). The microcrystalline tests carried out on these samples showed positive results for both BZP and caffeine without interference from other substances present.

KEYWORDS Mephedrone, MDAI, benzylpiperazine, legal high, microcrystalline test, designer drugs

INTRODUCTION
The internet has recently been flooded with new synthetic recreational drugs, so called 'legal highs', easily available for everyone to buy. Substances are commonly advertised as fertiliser for plants, research chemicals or bath salts but are being used to replace more strongly controlled psychoactive stimulants like MDMA or cannabis. The UK is continuously banning the legal use of novel emerging designer drugs and controlling them under the different amendments of the Misuse of Drugs Act 1971 [1-3].
In 2010 various publications investigated the homogeneity of internet bought samples [4-6] and revealed that on many occasions the advertised active ingredient was not actually sold. Instead, often dangerous cocktails of illicit drugs bulked out with legal stimulants like caffeine were purchased. Recently banned substances like 4-methylmethcathinone (mephedrone), benzylpiperazine (BZP) and 1-[3-(Trifluoromethyl)phenyl]piperazine (3-TFMPP) have been found in supposedly legal 'highs'. Second generation legal highs like 5,6-methylenedioxy-2-aminoindane (MDAI) or 5-iodo-2-aminoindane (5-IAI) emerged after the bans of 2009 and '10 and were still legally available in the first half of 2011 [Figure 1]. Characterisation and analysis of novel drugs is difficult since adequate reference standards are often not easily available [6]. Given recent trends in the legislation regarding legal highs, second generation substances could be banned in the near future.
Publications on quick and simple non-instrumental analyses such as colour or microcrystalline tests for these new drugs are sparse. It has been reported that existing colour tests are not sensitive enough for some of the new drugs [7,8]. A colour test to detect mephedrone using Zimmermann's reagent as well as a simple TLC procedure using ninhydrin reagent for visualisation have been communicated [9]. Stimulants like BZP and 3-TFMPP have been analysed using well established colour test reagents, such as Simon's and Marquis', which are widely used by drug enforcement investigators [7]. The colour responses were reported to be similar to those of other stimulants like the amphetamines. Similarities to existing compounds are not surprising since a change in colour is always caused by a functional group rather than the molecule as a whole. Consequently, tests can be inconclusive and results easily misinterpreted.
Microcrystalline identification of drugs of abuse has been used for a long time in forensic drug analysis. The simplicity of the test layout paired up with the uniqueness of the results obtained provides a powerful tool when analysing unknown substances. Microcrystalline tests are direct tests as the analyte molecule itself becomes a part of the crystal structure and therefore a specific crystal habit is observed for many compounds [10].

It has been previously reported that compounds other than the drug of interest present in the tested sample can influence the microcrystalline test result [11,12]. Cutting agents like caffeine can crystallise with the reagent or hinder crystallisation and therefore result in distortion of the overall microcrystalline test appearance. In a recent publication Nelson et al. presented results were the effects of the adulterants caffeine and lidocaine on developing cocaine tetrachloroaurate microcrystals were investigated [12]. It was reported that an increasing adulterant concentration influenced the cocaine-reagent crystals by changing their appearance. Furthermore, differences in crystal habit were detected depending on the adulterant added which allowed some sort of identification of cutting agent present in a model street cocaine hydrochloride sample.

In this paper a simple microchemical identification test for mephedrone, BZP and MDAI using mercury chloride as a reagent is proposed. Mercury chloride has been reported and used as a reagent for microcrystalline tests to detect drugs of abuse like methadone and diamorphine [13,14]. Tests were developed and carried out using standards. Results were confirmed using internet bought samples, the composition of which had been analysed and published previously [6].



5,6-Methylenedioxy-2-aminoindane (MDAI)

Benzylpiperazine (BZP)

4-Methylmethcathinone (mephedrone)

Caffeine

Figure 1
Structures of selected designer drugs and one cutting agent

MATERIALS AND METHODS

Chemicals and Materials

Mephedrone hydrochloride (> 98.5 %) was purchased from ReseaCHEM (Burgdorf, Switzerland), 5,6-Methylenedioxy-2-aminoindane freebase (99.3 %) was from LGC GmbH (Luckenwalde, Germany), 1-benzylpiperazine freebase (> 97 %) was from Fluka (Steinheim, Germany). 1-[3-(Trifluoromethyl)phenyl]piperazine hydrochloride, cocaine hydrochloride, ephedrine hydrochloride (99 %), procaine hydrochloride, (±)-3,4-methylenedioxymethamphetamine hydrochloride (MDMA), D-amphetamine sulphate, DL-amphetamine sulphate, L-amphetamine freebase, (+)-methamphetamine hydrochloride, (±)-methadone hydrochloride and caffeine were purchased from Sigma-Aldrich (Gillingham, UK). (-)-Ephedrine sulphate (98 %) was from Acros Organics (New Jersey, USA). Mercury dichloride (> 99.5 %) and mercury (II) iodide (> 99 %) were from Sigma-Aldrich. All water used was deionised to 18.2 MΩ resistance using an Elga Labwater Purelab flex system (High Wycombe, UK). Methanol (HPLC-grade) was obtained from Fisher Scientific (Loughborough, UK).

Four legal high samples were purchased online in August 2010 as MDAI, 5-IAI, Benzo fury and NRG-3 including a free sample of E2 from *www.benzofury.me.uk*; two further MDAI labelled samples were purchased in October 2010 from *www.VIPlegals.com* and *www.wide-mouth-frog.com*.

Microcrystalline tests were carried out on standard microscope glass slides purchased from Fisher Scientific and used as obtained.

Microcrystalline test method

Drug standards were prepared as aqueous solutions at concentrations of 10 g/L for mephedrone and 1 g/L for MDAI and BZP. MDAI was dissolved in methanol first and then diluted in water resulting in an overall solvent content of 10 % methanol. Other psychoactive stimulants were tested by using a few grains of the drug standard dissolved in 10 µL water on a microscope slide.

The reagent was an aqueous solution of mercury chloride at a concentration of 10 g/L. Tests on MDAI were carried out with 10 g/L mercury chloride solutions containing 10 % methanol. Mercury iodide was dissolved in 100 % methanol as were the drug standards tested with it.

Microcrystalline tests were set up by mixing 10 µL of drug solution with 10 µL of the reagent on a glass slide. Nucleation was assisted and therefore crystallisation encouraged by gently swirling a plastic pipette tip a few times within the freshly mixed drop. After the initial encouragement tests were not further disturbed.
Drug and reagent controls were carried out alongside all experiments by applying the respective volumes of drug or reagent solution only to slides, assisting nucleation with a pipette tip and observing crystallisation patterns throughout and at the end of the test.
Progress of the microcrystal development was observed with a Meji ML 5000 microscope (Axbridge, UK) under low and high magnification using calibrated transmitted light microscopy and phase contrast microscopy set for Kohler illumination at 400x overall magnification.
Images were taken using a Canon EOS Rebel T2i digital SLR camera fitted with a Meiji Techno adapter ring, camera attachment and 1.9 x magnification photo eye piece.

RESULTS & DISCUSSION
The reaction between mephedrone, MDAI, BZP and mercury chloride was carried out independently. Descriptions of the obtained crystals are listed in table 1 along with the results of mercury chloride combined with other stimulants.

The mephedrone test
The mephedrone-reagent crystals formed paddlewheel clusters consisting of several square-cut blades [Figure 2a]. Crystal clusters were three-dimensional shapes that grew in length, depth and height within the dimensions of the applied droplets. The speed of growth varied between 1 and 30 minutes depending on the drug concentration tested, with an increasing crystal growth rate as concentration increased.
It appeared that crystallisation consistently originated from a central point of nucleation. Crystal clusters were denser and less translucent when nucleation was unassisted, whereas when nucleation was assisted crystals appeared faster and in a more organised manner developing along the movement lines forming chains of blades like barbwire. Then the central point of nucleation was of longitudinal dimension and only a few paddlewheel clusters could be detected.
It was also observed that too vigorous movement during assisted nucleation would disturb the orientation of the densely packed chain, nuclei would be separated from the chain and single blades of varying dimensions would grow.
At concentrations lower than approximately 5 g/L fewer crystals formed. Crystal clusters were sparse and single blades were more common. Premature paddlewheel structures consisting of only a few pointy plates appeared within the periphery of the drop.
It was important to observe the test periodically during the development as mephedrone formed drug-only crystals easily when in excess covering already formed drug-reagent crystals which made the identification of mephedrone increasingly difficult.
Mephedrone controls crystallised covering the test area as transparent scales of joint plates which had no resemblance to the mephedrone-reagent crystals.

The MDAI test
The MDAI freebase standard was not soluble in water and therefore the substance was dissolved in methanol and subsequently diluted in water resulting in an overall methanol content of 10 % (v/v). The mercury chloride reagent used to develop the MDAI tests was also prepared in 10 % (v/v) methanol to prevent precipitation before forming microcrystals. The low percentage of methanol did not speed up the evaporation of the test significantly and crystallisation was not hindered.
MDAI freebase and mercury chloride formed flat serrated blades of varying dimensions, which became more irregular with increasing size [Figure $2b_i$]. Smaller crystals would grow as single blades and larger crystals would develop as predominantly two-dimensional bunch structures which would spread out fanwise from a single joining-point. Small clusters of single or joint blades would break off while the test drop was still wet. Upon drying larger blade structures would dehydrate leaving straw-like crystal bunches which could be unequivocally identified at low magnifications despite the distortion.
Crystals grew within 60 seconds after assisted nucleation along the trail the pipette had left but would also appear over time in the non-assisted regions of the drop.
MDAI freebase controls showed large irregular transparent plates of drug crystals forming as crusts from the edges of the drop.
At the time of conducting this study an MDAI hydrochloride standard was not available. MDAI freebase was converted into its hydrochloride salt by adding concentrated hydrochloric acid to MDAI solutions. After thoroughly mixing for 20 seconds using a vortex mixer tests were carried out using aqueous mercury chloride reagent solutions. After a couple of minutes trapezoidal blades grew within the test area [Figure $2b_{ii}$]. MDAI hydrochloride controls crystallised as random skeletons covering the test area.

Overall, MDAI freebase and MDAI hydrochloride resembled each other by forming crystal blades when mixed with mercury chloride.

The benzylpiperazine test

BZP and mercury chloride formed transparent flat square-cut plates. Towards the edges the crystals affected the passage of light waves which created an appearance of colour. The feature was enhanced when observing the BZP-reagent microcrystals using phase contrast microscopy [Figure 2c].

A concentration of 10 g/L of BZP resulted in instantaneous white precipitation when mixed with the reagent masking any potential crystal formation. A ten fold dilution enabled the detection of microcrystals as seen in figure 2c. Nucleation was assisted but if the motion was carried out too vigorously, crystallisation would turn into a rapid precipitation of drug and reagent hindering observation of crystals. At lower concentrations the force of assistance had less impact. When nucleation was not assisted fewer square-cut plates grew and were masked by a thin layer of crystal scales which made identification more difficult compared to assisted nucleation.

Crystals grew within 30 seconds after assisted nucleation on the trails of the pipette swirls. After a couple of minutes crystals would appear anywhere within the test area but not necessarily originating from the edge of the drop. At the tested concentrations crystals could be identified with confidence even when the test was fully developed and the area dried out.

The size and quantity of the crystals appeared to be related to the concentration of BZP. When the concentration was decreased, approaching the limit of detection, significantly fewer but larger crystals would grow. Some large square plates could be easily spotted at lower magnifications despite excess reagent crystals.

BZP controls would grow as densely packed, mainly undefined bushes covering the whole test area by forming a crusting layer of irregular crystallisation.

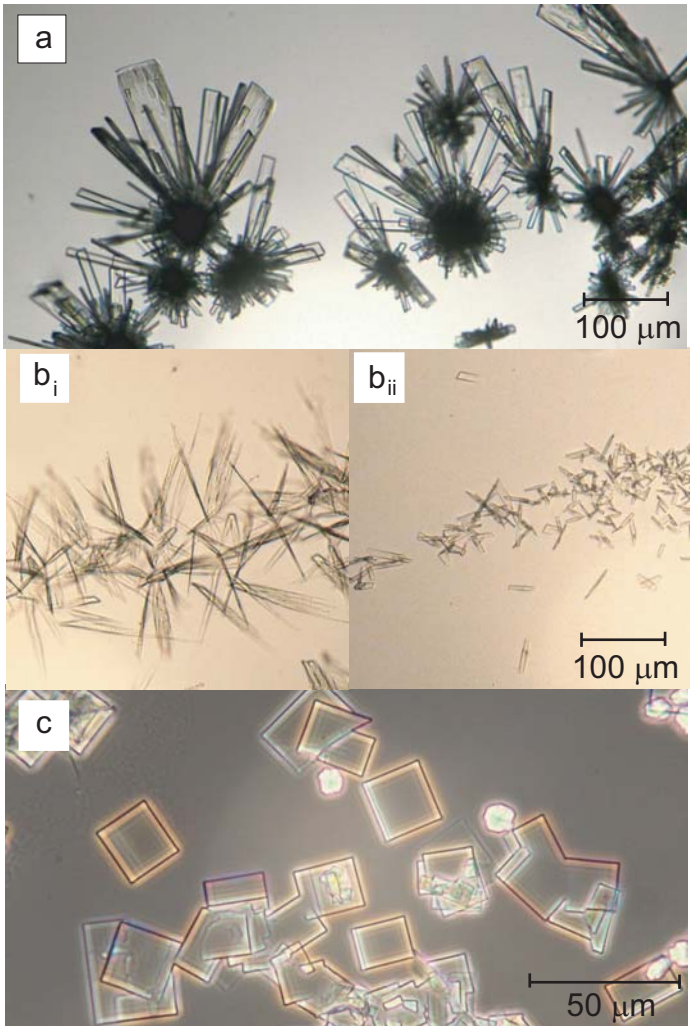


Figure 2
Microcrystals formed with mercury chloride and a) mephedrone (c = 10 g/L), b_i) MDAI freebase (c = 1 g/L), b_{ii}) MDAI hydrochloride (c = 1 g/L) and c) BZP (c = 1 g/L)

Confirmation of the uniqueness of the test

Structurally similar drugs as well as other psychoactive stimulants were tested using the mercury chloride reagent to confirm the uniqueness of the above described crystals. All resulting crystal responses are summarised in Table 1 and a few selected shown in Figure 3.

The test results did not show crystal patterns similar to those obtained with mephedrone, MDAI and BZP. The observed crystal structures for all other tested substances were in accordance with the results reported by Fulton and Clarke in 1969 [13,14]. The microcrystalline response of 3-TFMPP, MDMA and γ-hydroxybutyric acid with mercury chloride has not been published previously. At higher concentrations some drugs showed similar looking crystallisation patterns which may be explained by the structural similarity of the tested drugs.

Table 1
Microcrystal description of selected psychoactive stimulant drugs tested with mercury chloride

Drug	Crystal description	Cross reference Figure 2
3-TFMPP	white precipitation, no crystal formation	-
BZP	square plates	a
Caffeine	long needles	b
Cocaine	no crystal formation	-
D-Amphetamine	dense rosettes and bunches of hair like needles	c
DL-Amphetamine	smudge rosettes	d
Ephedrine hydrochloride	skeletonised crosses	e
Ephedrine sulphate	bunches of hair like needles	f
γ-hydroxybutyric acid	no crystal formation	-
L-Amphetamine	white precipitation with some plates	-
MDAI	bunches of serrated blades	g
MDMA	no crystal formation	-
Mephedrone	paddlewheels and rosettes of blades	h
Methamphetamine	skeletonised and branched rods and plates	i
Procaine	no crystal formation	-

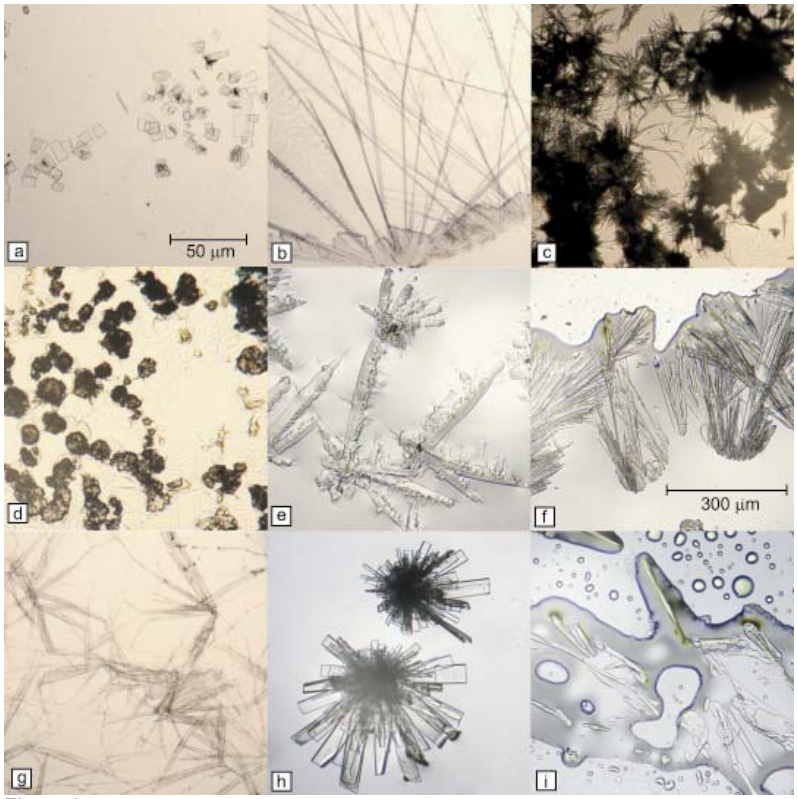


Figure 3

Microcrystalline response of selected psychoactive stimulant tested with mercury chloride as reagent; a) BZP, b) caffeine, c) D-amphetamine, d) DL-amphetamine, e) ephedrine hydrochloride, f) ephedrine sulphate, g) MDAI, h) mephedrone, i) methamphetamine.
Footnote: image a) was taken at 76 x magnifications, images b) to i) were taken at 19 x magnifications.

Limit of detection for the microcrystalline test

The limit of detection was determined by approaching the concentration where no specific drug-reagent crystals would form. Cut-off values were set at the lowest concentration tested which unmistakably resulted in drug-reagent crystals as recommended by the SWGDRUG [15]. The concentration range analysed was 10 g/L to 0.1 g/L for mephedrone and 1 g/L to 0.1 g/L for MDAI and BZP. Repeats of $n = 18$ samples originated from three different stock solutions and were carried out alongside numerous reagent and drug controls to ensure accuracy of results.

A grading system was developed to evaluate test results consistently [Table 2]. Each grade was allocated to be a positive or negative response. The crystals in the description refer to drug-reagent crystals only and do not include excess reagent crystallisation. The grading system was developed based on numerous repeats of various microcrystalline tests. There was no definite way to point out differences between neighbouring grades but an overall subjective impression was used to define the grades for the limit of detection study.

In order to evaluate the limit of detection and to check for potential trends the results were plotted in a diagram of test response over number of experiments [Figure 4]. Grades of 4 and 5 were considered as true positives. The awarding of grade 3 was dependant on the test carried out. For example the mephedrone microcrystalline test always presented in either the one extreme of plenty of well-formed crystals or the other of nothing identifiable at all; BZP could only have a few well-formed crystals which were easily rated as a true positive due to the perfect habit of the crystal.

Grades 1 and 2 were rated as negative as the presence of distorted crystals hinders correct identification of the sample when performing microcrystalline tests. Grade 0 was evaluated to be a true negative because of the absence of crystals.

Table 2
Grading system for evaluating the limit of detection of microcrystalline tests

Grade	Positive (P)/Negative (N)	Description
5	P	numerous well-formed crystals
4	P	fair amount of well-formed crystals
3	P	few well-formed crystals
2	N	few distorted crystals
1	N	almost no crystals (if present distorted)
0	N	no crystals

The mephedrone diagram [Figure 4a] shows a clear distinction of the given grades at concentrations of 5 g/L and 2.5 g/L. Only little distinction could be seen between concentrations of 2.5 g/L and 1 g/L since some negative results (grade 0) with no crystal growth were detected at 2.5 g/L. Results which were graded as 1 and 2 were evaluated as negative despite crystal formation as they presented distorted and therefore could not be easily identified as unique mephedrone-reagent crystals.

Overall, a clear distinction between the three tested concentrations could be made. The limit of detection could be narrowed down to approximately 3 g/L. At 2.5 g/L a few distorted crystals could be detected but identification as a positive test was borderline and somewhat difficult carried out by an inexperienced user. Based on these results the cut-off value for the mephedrone microcrystalline test was set to be 5 g/L as tests always gave true positive results. At a concentration of 1 g/L and below no mephedrone-reagent crystals formed [Table 3].

The optimum concentration for mephedrone crystal formation was around 10 g/L when using the mercury chloride reagent at a concentration of 10 g/L. Plenty of drug-reagent crystals formed within a couple of minutes when assisting the nucleation. If mephedrone is suspected in a sample but the above described crystals can not be obtained, it is recommended to test higher and lower concentrations of the sample solution as well as the powdered sample directly.

The MDAI diagram [Figure 4b] shows a fairly clear distinction between true positive and negative results. Concentrations of 0.5 g/L always gave positive results being rated between 4 and 5 on the grading system. At 0.4 g/L crystals would either grow in vast quantities and of perfect habit resulting in scores of 4 and 5 or in 10 out of 18 tested times no crystal growth at all could be observed. Concentrations of 0.3 g/L were clearly detected as negatives rated between 0 and 2. Based on the scores given the limit of detection and the cut-off value were set at 0.5 g/L [Table 3]. The optimum microcrystalline test concentration for MDAI was at 1 g/L as within a short time window plenty of large crystals grew upon assisted nucleation. This test can be easily evaluated when wet or dry but the dehydration of crystals resulting in a slightly different crystal habit needs to be taken in account.

The BZP diagram [Figure 4c] presented differently from the other tested drugs. No clear distinction between the tested concentrations could be seen. At concentrations of 0.3 g/L crystals of perfect quality would grow in large quantities. The results at a concentration of 0.2 g/L were unique from other microcrystal tests as very few but comparably large crystals in perfect habit would grow. At a concentration of 0.1 g/L the characteristic square crystals were absent but sometimes distorted and irregular flat plates could be observed. The limit of detection was determined to be 0.2 g/L and the cut-off value was set to be 0.3 g/L [Table 3]. The optimum microcrystalline test concentration for BZP was at 1 g/L.

The evaluation of the limit of detection was somewhat subjective as to whether specific drug-reagent crystals had formed at critical concentrations around the limit of detection. At lower concentrations crystals formed much slower and often distorted in shape and habit. In order to keep results consistent it was decided to observe all microcrystalline tests when completely dry. However, this introduced the possibility of misinterpretation due to the masking of crystals of interest by excess reagent crystallisation or distortion of crystals based on dehydration of the test area.

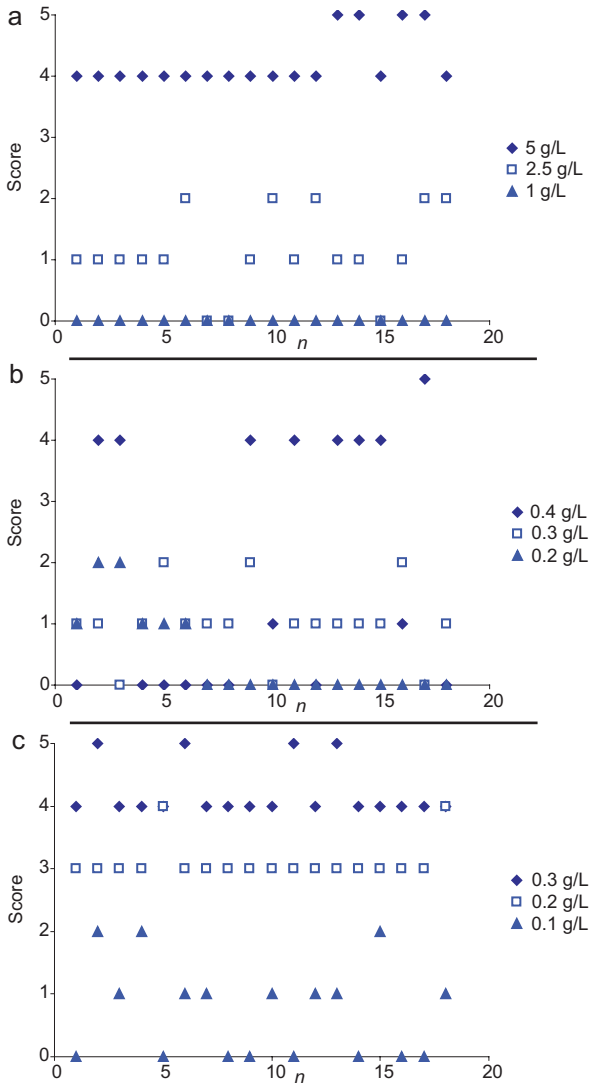


Figure 4
Graphic evaluation of the limit of detection for a) mephedrone, b) MDAI and c) BZP using microcrystalline testing with mercury chloride as reagent

Table 3
Limit of detection and cut-off value for microcrystal detection of mephedrone, MDAI and BZP using the mercury chloride reagent

Substance	Limit of detection (g/L)	Cut-off (g/L)
Mephedrone	3.0	5.0
MDAI	0.5	0.5
BZP	0.2	0.3

The caffeine study

The effects of caffeine, as a cutting agent, on microcrystal development were investigated. Caffeine formed long needles when mixed with mercury chloride [Figure 3b]. The impurity of street samples was modelled by mixing volumes of caffeine and drug solutions on the slide (c = 1 g/L for BZP and MDAI, c = 10 g/L for mephedrone; c = 1 g/L for caffeine) before adding the reagent; nucleation was assisted gently.

Mephedrone, MDAI and BZP were tested in proportional content ranging from 10 to 90 % (v/v). Mephedrone crystals could be observed from 50 % (v/v) and greater mixed with caffeine, MDAI from 40 % (v/v) and BZP from 20 % (v/v) [Table 4]. Caffeine crystals formed in all tested mixtures and could be easily identified. Only at MDAI percentages of 40 and above could the caffeine needles appear slightly distorted when being covered or joined with MDAI crystals. Since the habit of both MDAI- and caffeine-mercury chloride crystals is very different the deformation of crystals from the standard compound appearance did not influence the unequivocal identification of either substance.

These results were comparable to tests conducted by ATR-FTIR on a solid MDAI standard mixed with caffeine at various percentages [6]. The dominance of caffeine peaks at 90% (w/w) disguised MDAI features in the spectrum in a way that identification of the active ingredient was impossible.

Table 4
Results of positive microcrystalline response for mephedrone, MDAI and BZP when altered with caffeine

Substance	Drug level necessary for positive identification (% v/v)
Mephedrone	≥ 50
MDAI	≥ 40
BZP	≥ 20

The mercury chloride reagent

Mercury chloride controls crystallised in random three-dimensional grains and tablets. Microcrystalline tests where drug-reagent crystals predominantly grew within the periphery of the drop were likely to show some reagent-only crystals on the drying solvent front, especially at drug concentrations towards the limit of detection. Tests where drug-reagent crystals mostly originated from the border of the drop, reagent-only crystal formation was delayed until the test drop was about to dry out.

In order to confirm that observed microcrystals were only formed when mercury (II) ions were present tests with mercury (II) iodide were carried out. Unlike the chloride, the iodide did not dissolve well in water with a solubility of 7.4 g/100 mL at 20°C and solutions were therefore prepared in 100 % methanol. All drugs tested with mercury iodide were prepared in methanol to avoid precipitation of the reagent on contact with water.

Microcrystalline tests are usually not carried out in solvents with a boiling point below 100°C as the rate of evaporation is too fast for crystals to develop to a decent size for certain identification. However, in order to determine whether the cation or anion is taking part in the crystal formation process, prematurely developed crystals were acceptable for comparison.

Mephedrone formed bunches of colourless short needle rosettes when exposed to mercury iodide. Crystals did not develop into blades but overall resembled the three-dimensional structures observed when aqueous solutions of the drug is exposed to mercury chloride. MDAI produced serrated blades which developed to a smaller size compared to the aqueous mercury chloride reagent. Caffeine presented in needles which grew somewhat skeletonised and were very similar to the mercury chloride reagent. Only BZP produced too premature crystal structures of random grains to be judged a true positive.

The similarity in crystal habit of 3 out of the 4 tested substances confirmed that indeed the mercury (II) ion takes part in forming the above described specific microcrystals. The detected changes in crystal habit were potentially related to the change of solvent rather than the associated halogen anion.

Application to internet purchased samples

Seven legal high samples were purchased from three different websites in the second half of 2010 under the names of MDAI (3 products), 5-IAI, Benzo fury, NRG-3 and E2. Aqueous solutions (c = 1 g/L) of all 7 internet samples were tested with the mercury chloride reagent. Test drops were gently assisted for nucleation.

Microcrystals for BZP and caffeine formed in two of the three MDAI samples as well as the Benzo fury, 5-IAI and NRG-3 products. The E2 sample produced simply caffeine crystals and only one of the MDAI samples formed the MDAI specific serrated blades. The absence of other crystals highlighted the reliability of the investigated microcrystal tests. Any other cutting agents present in the internet samples did not interfere with development of the drug-reagent crystals which in all cases formed first.

Methanol extracts of these samples were analysed by FTIR-ATR and GC-MS and results were published early 2011 [6]. The microcrystalline test results were in accordance with the findings ascertained from the analytical techniques [Table 5]. The presence of 3-TFMPP and potentially other unidentified substances did not hinder the crystal formation of MDAI, BZP and caffeine in the respective samples.

Table 5
Summarised results of the internet legal high samples purchased in the second half of 2010

Product	Website	Active substances identified by microcrystalline test	Active substances identified by FTIF/GC-MS
MDAI	www.benzofury.me.uk	BZP caffeine	BZP caffeine 3-TFMPP
Benzo fury	www.benzofury.me.uk	BZP caffeine	BZP caffeine 3-TFMPP
5-Iodo-2-aminoindane (5-IAI)	www.benzofury.me.uk	BZP caffeine	BZP caffeine 3-TFMPP
NRG-3	www.benzofury.me.uk	BZP caffeine	BZP caffeine 3-TFMPP
E2	www.benzofury.me.uk	caffeine	caffeine
MDAI	www.VIPlegals.com	MDAI	MDAI
MDAI	www.wide-mouth-frog.com	BZP caffeine	BZP caffeine 3-TFMPP

CONCLUSION

A rapid detection of mephedrone, MDAI and BZP using microcrystalline tests has been developed. Upon mixing with aqueous mercury chloride as a test reagent each drug formed specific microcrystals which were observed using transmitted light and phase contrast microscopy. Microcrystalline tests are versatile and can be carried out in as little as 10 minutes by dissolving a few granules of sample in 10 µL water on a slide, adding 10 µL of reagent and waiting a couple of minutes for crystals to develop. Detection via a microscope in less than a minute and comparing with crystals obtained using drug standards make microcrystalline tests a valuable contender in the race for rapid identification of seized samples. Moreover, no expensive and high maintenance analytical technology is needed to carry out successful tests.

Tests with the cutting agent caffeine revealed that the specific microcrystal formation was not hindered for any of the investigated substances. The results were confirmed on internet bought samples of legal highs. BZP was detected alongside caffeine in mixtures also containing 3-TFMPP. Neither of the specific crystals were masked or influenced by the presence of other compounds in the mix.

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