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# PRESUMPTIVE TESTING OF AMPHETAMINE-TYPE STIMULANTS *VIA* COLOUR TESTS

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## ABSTRACT

This paper focuses on the use of colour tests to identify the possible presence of amphetamine-type stimulants (ATS). At present in Brunei Darussalam, some of the screenings for the presence of ATS in the Narcotics Laboratory make use of test-kits which are expensive. In this paper, various test reagents such as Chen, Simon, Marquis, Mandelin, Mecke and Froehde reagents were prepared.[6,7] Except for Mandelin reagent, these test reagents were found to be stable after a month. The concentrations of samples have an effect on the colour changes. All reagents, except for the Mandelin reagent, gave good and reliable results. This therefore provides a fast and economical way of initial screening for ATS.

*Keywords:* presumptive tests, amphetamine-type stimulants, colour tests

## INTRODUCTION

Amphetamine-type stimulants (ATS) are drugs that are structurally derived from β-phenethylamine (β-PEA) (see Figure 1) and examples include amphetamine, methamphetamine, phentermine, 3,4-methylenedioxyamphetamine (MDA), 3,4-methylenedioxyethylamphetamine (MDEA), 3,4-methylenedioxymethamphetamine (MDMA) and many more. They are easy to produce, cheap to buy and hard to control. They can be more potent than cocaine and usually have a longer-lasting effect.

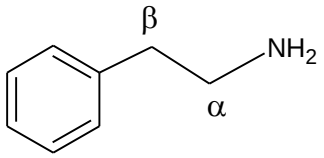


Figure 1: β-PEA

ATS have been used as far back as the Second World War where fighter pilots and soldiers were given drugs such as amphetamines to keep them awake. In fact, many of these ATS drugs were synthesized for medical purposes. However, in recent years there has been a prominent increase in the production and use of ATS worldwide. These drugs can be totally synthetic, and indeed, precursor chemicals are subject to separate controls and monitoring. As a result, instead of using ATS for medicinal purposes, due to their ease of synthesizing them and their stimulant effects on the users, these drugs have gained popularity in the illegal black markets and pose serious drug trafficking problems throughout the world.

According to reports by United Nations International Drug Control Programme (UNDCP) there is now an increase in the shift of drug trafficking from classic drugs such as heroin to synthetic ATS drugs. Make-shift clandestine laboratories can be located anywhere from one's backyard to warehouse and so on. These laboratories are on the rise in East and South East Asia especially in countries such as Myanmar, Philippines, Malaysia, Indonesia, Cambodia and China.[1,2] One of the largest amphetamine laboratories was found in Guangdong, China, where authorities seized more than 1.7 metric tones of liquefied methamphetamine.[3]

The drugs themselves are mixed with a wide variety of adulterants and diluants and also contain a cocktail of related products and impurities from the manufacturing process. The purpose of adding adulterants and diluants are either to hide the lack of the desired product, dilute it, and add another type of effect to the drug mixture or to improve the properties of the tablets prior to the tabletting process.[4]

In 1998 the Government of Brunei Darussalam upgraded methylamphetamine from a class B to class A drug. Drug traffickers caught with 50 g or more of methylamphetamine will face a mandatory death penalty.[5]

Presumptive tests are designed to provide an indication of the presence or absence of drug classes in the test sample. As stated in the United Nations Office on Drugs and Crime (UNODC) manual[6], there are three types of presumptive tests - colour tests, anion tests and microcrystal tests.

Colour test kits which are available are usually very costly. This paper focuses on the potential use of various reagents, which were prepared in the laboratory, to identify possible ATS using colour tests from both the UNODC's and Clarke's procedures on the selected ATS stock solution and solid sample. By using a series of the reagents, one can determine the possible type of drug base on the change in colour for selected ATS as specified by Clarke's[7], UNODC[6] and EZ test kit[8]. This will also provide a cheaper and more economical way for quick screening for ATS.

## METHODOLOGY

### *Materials and methods*

All chemicals used were purchased from Aldrich-Sigma, Ajax. A total of eight ATS were used *i.e.* amphetamine, d-methamphetamine, phentermine, MDMA, MDEA, ketamine, *N*-propylamphetamine and MDA. All ATS stock solutions were prepared in methanol. All reagents and colour tests were carried out using the UNODC's procedure and the procedure as stated in "Clarke's Analysis of Drug and Poisons".[6,7]

### *Test Reagents*

The reagents used for colour tests were Chen's Reagents, Simon Reagent, Marquis Reagent, Mandelin Reagent, Mecke Reagent and Froehde Reagent.

## RESULTS AND DISCUSSION

### *Colour tests on the stock solutions*

The change in colours for the stock solutions using the various prepared reagents is shown in Table 1. By comparing with the reported colour change shown in Table 2,[6,7,8] only Simon, Chen, Marquis and Mecke reagents showed consistent result with that of the literature. Simon's test is generally used as a test for the presence of methamphetamine, MDA, MDMA, MDEA and other secondary ring-substituted amphetamines. Mecke's reagent provides a useful presumptive test for opium alkaloids and some ATS while Chen's test is used to distinguish ephedrine, pseudoephedrine, norephedrine, phenylpropanolamine and methcathinone from amphetamine and methamphetamine.

Marquis reagent is the most common drug testing kit base reagent and has been used as an indicative test by forensic and law enforcement bodies for many years. Currently, it is used for presumptive testing for morphine, heroin and amphetamine. However, it is not

authorized as a presumptive test for ecstasy as it can only give an indication. Marquis reagent alone cannot identify ketamine or *para*-methoxyamphetamine (PMA) or distinguish between MDA, MDEA, and MDMA. Using Mandelin reagent in conjunction with Marquis does increase the accuracy of the test and appears to indicate PMA and ketamine[9].

With Froehde reagent there was no visible reaction being observed for d-methamphetamine initially but a light orange solution was observed on standing. Even though Froehde reagent failed to give a positive test for d-methamphetamine, nevertheless majority of the other ATS drugs when tested with Froehde reagent gave good correlations with that reported in literature. Hence Froehde reagent can overall be considered a reliable reagent.

**Table 1: Colour change for the selected ATS stock solution using the prepared reagents**

| **Substances** | **Mandelin** | **Marquis** | **Simon** | **Mecke** | **Froehde** | **Chen** |
|---|---|---|---|---|---|---|
| Amphetamine | No reaction* | Light orange | No reaction* | Light yellow | No reaction to pale yellow | No reaction* |
| d-Methamphetamine | No reaction* | Light orange | No reaction* | No reaction | No reaction to light orange | No reaction* |
| Phentermine | No reaction* | Light orange | No reaction* | No reaction | Light brown | No reaction* |
| MDA | No reaction* | Light purple to black | No reaction* | Dark blue | Brownish green | No reaction* |
| MDMA | No reaction* | Light purple to black | Deep blue | Dark blue | Brownish green | No reaction* |
| MDEA | No reaction* | Light purple to black | light blue | Dark blue | Brownish green | No reaction* |
| Ketamine | No reaction* | No reaction | No reaction* | No reaction | No reaction | No reaction* |
| *N*-Propylamphetamine | No reaction* | No reaction | Light blue | Pale yellow | No reaction | No reaction* |

*Note:* * the colour of the reagent is considered as negative

**Table 2: Expected colour change for the selected ATS with respective reagents**

| Substances | Reagents | | | | | |
|---|---|---|---|---|---|---|
| | **Mandelin** | **Marquis** | **Simon** | **Mecke** | **Froehde** | **Chen** |
| Amphetamine | Dark Green or brown | Orange to brown | Pink to cherry red or no reaction | Yellow or no reaction | Colourless | No reaction |
| Methamphetamine | Dark Green | Red or orange to brown | Deep blue | No reaction | No reaction | No reaction |
| Phentermine | Green | Light orange | - | Light orange | Brown | No reaction |
| MDA | Dark purple | Dark purple | Pink to cherry red or no reaction | Dark blue or dark purple | Green to dark violet | No reaction |
| MDMA | Dark purple | Dark purple | Deep blue | Dark blue or dark purple | Yellow to dark blue | No reaction |
| MDEA | Dark purple | Dark purple | Deep blue | Dark blue or dark purple | - | No reaction |
| Ketamine | Orange or brown | No reaction | No reaction | No reaction | No reaction | No reaction |

*Note:* * the colour of the reagent is considered as negative

Mandelin reagent did not give any positive result on all the stock solutions and this finding is in contrast to what was expected (Table 2). Mandelin reagent was supposed to show different colours for MDA, MDMA, MDEA, ketamine, methamphetamine and PMA. However, when tested Mandelin Reagent did not show any changes in all the stock solutions. Various reasons could attribute to this. For example, the ammonium vanadate used might have already degraded or it could be that the reagent is not stable since it turned from an orange solution to yellow precipitate after ten days. Alternatively, it might due to the solvent effect which alters the reaction.

The colour tests were therefore repeated using freshly prepared Mandelin reagent. Only MDA, MDMA, MDEA and *N*-propylamphetamine showed colour change to green, pale green and yellow respectively. Amphetamine and methamphetamine gave very slight colour change from orange to dark yellow. Even though there were colour changes, none of these colour changes correspond with the expected colour changes in Table 2. This could

be due to either the concentration of the stock or impurities presence as the stock solution is in its salt form. The newly prepared Mandelin reagent was then tested on solid methamphetamine and ketamine. Greenish blue solution was observed for methamphetamine while reddish brown for ketamine. Since the colour of the reagent itself is orange, the colour change could hardly be seen. However, as expected, the colour change was more distinct when solid sample was used.

***Stability of the Reagents***

In order to study the stability of the reagents, the same screening was repeated after one month using the stock solutions. The only exception was the Mandelin reagent which was prepared two weeks before this repeated screening. From the result (Table 3) it can be deduced that the prepared reagents were still stable after one month except for Mandelin Reagent. It can then be assumed that the Mandelin Reagent was not stable. Hence it is recommended to prepare this reagent freshly.

**Table 3: Colour changes for the selected ATS stock solutions using a month old reagents**

| Substances | Mandelin# | Marquis | Simon | Mecke | Froehde | Chen |
|---|---|---|---|---|---|---|
| Amphetamine | No reaction* | Light orange | No reaction* | Light yellow | No reaction to pale yellow | No reaction* |
| d-Methamphetamine | No reaction* | Light orange | No reaction* | No reaction | No reaction to light orange | No reaction* |
| Phentermine | No reaction* | Light orange | No reaction* | No reaction | Light brown | No reaction* |
| MDA | No reaction* | Light purple to black | No reaction* | Dark blue | Brownish green | No reaction* |
| MDMA | No reaction* | Light purple to black | Deep blue | Dark blue | Brownish green | No reaction* |
| MDEA | No reaction* | Light purple to black | light blue | Dark blue | Brownish green | No reaction* |
| Ketamine | No reaction* | No reaction | No reaction* | No reaction | No reaction | No reaction* |
| *N*-Propylamphetamine | No reaction* | No reaction | Light blue | Pale yellow | No reaction | No reaction* |

*Note:* * the colour of the reagent is considered as negative; # Reagent was two weeks old

***Colour tests on test sample***

A test sample, methamphetamine, was screened using Marquis, Chen's and Simon reagents and the solution turned dark brown in Marquis Reagent, deep blue to black in Simon Reagent while there was no change in colour for Chen's test. By comparing with the expected results shown in Table 2, it can be deduced that the test sample is methamphetamine.

For further confirmation, the test sample was screened using all the prepared reagents together with methamphetamine reference standard solution and control reagent. The results of the colour change are shown in Table 4 and Figure 2. All reagents gave the expected result for methamphetamine except for Froehde and Mecke reagents which gave yellow colours when thre was supposed to be no colour change. Nevertheless, from the tests it can then be concluded that the test sample is actually methamphetamine.

**Table 4: Colour change for the test sample using the prepared reagents.**

| **Test reagent** | **Observation** |
|---|---|
| Marquis | Orange to dark brown |
| Mecke | yellow |
| Froehde | yellow |
| Mandelin | Dark green |
| Simon | Deep blue |
| Chen | No reaction |

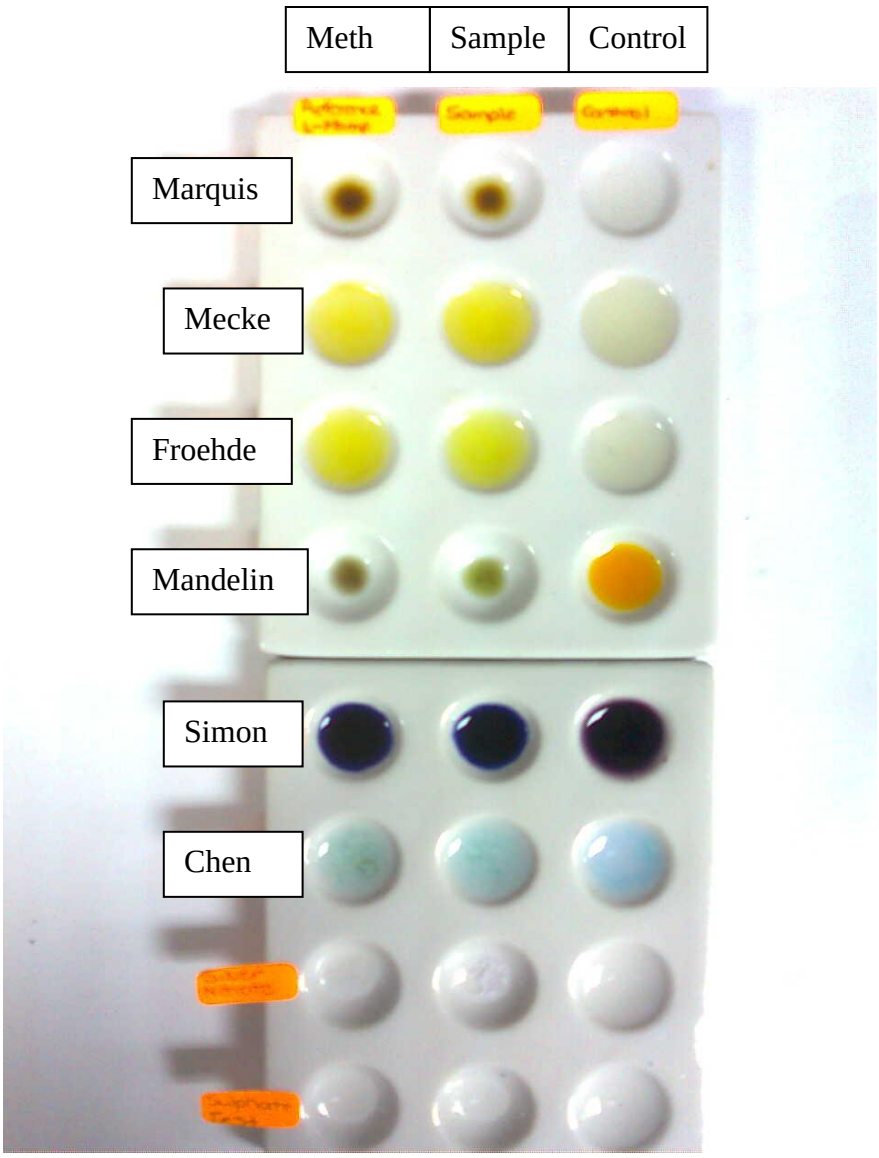


**Figure 2: Colour tests on a test sample**

***Effect of concentration in colour tests***

Simon Reagent was used to determine the effect of concentration on the intensity of the colour change. A series of different concentration methamphetamine solutions ranging from 0.1 to 1.3 mg/mL and solid sample were prepared. The result (Figure 3) showed that as the concentration decreases, the intensity of the colour also decreases. For the solid sample, a more intense colour was obtained compared to the liquid samples. This is in line with the solid sample having a higher concentration then liquid samples hence it will give the most intense and distinct colour change.

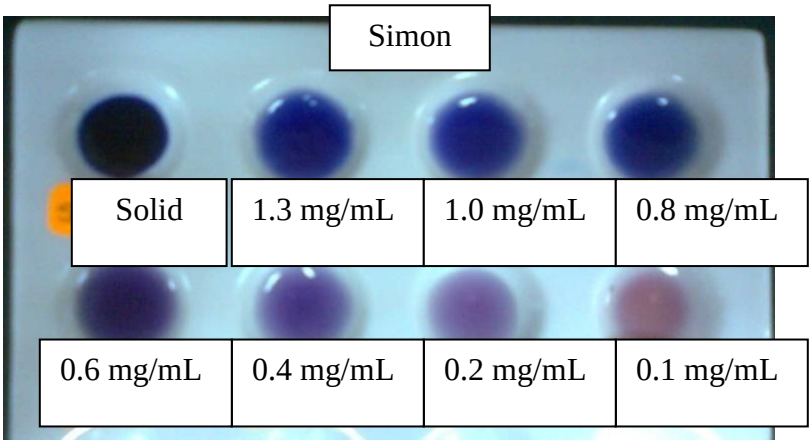


**Figure 3: Colours for different concentrations of test sample using Simon Reagent**

## CONCLUSION

Presumptive test does not provide any information on the purity of the test sample. It can only indicate the possible identity of the drug that is being analyzed. From the findings in this paper, concentration of test sample does affect the intensity of the colour change. Consequently, special attention should be paid during the validation process in particular on the determination of the limit of detection and the limit of quantitation.[11]

This study is a prelude replacing the current use of expensive test kits with cheaper, readily available reagents and the results are promising. Colour tests do not identify a drug but serve to narrow down the list of drugs possibly present in a sample. Result obtained from color test can only be served as the preliminary and presumptive evidence. Colours exhibited by these tests cannot be described with any accuracy due to the uncertainty in interpreting the colour change, concentration of the sample being analyzed and also the impurities presence.

Since presumptive tests are not considered sufficient for drug identification, results must be confirmed by additional laboratory tests. As pointed out by Pointer and Ali[10], the way to obtain accurate qualitative and quantitative information regarding all the contents of pills is to use laboratory based testing techniques such as thin layer chromatography, high pressure liquid chromatography and gas chromatography.

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## REFERENCES

1. Amphetamines and Ecstasy: 2008 Global ATS Assessment (United Nations Publication, Sales No. E.09.XI.12).

2. Country Report by the Indonesia (UNODC/HONLAP/2009/32/CR P.8). Thirty-second Meeting of the Heads of National Drug Law Enforcement Agencies, Asia and the Pacific (Bangkok, February 2009).

3. UNODC, Global SMART update 2009 (March), Volume 1.

4. Cole, M. D. (2003). *The Analysis of Controlled Substances.* Chichester, West Sussex, England: John Wiley & Sons.

5. The Misuse of Drug Act (MDA), Chapter 27, Drug Law, Negara Brunei Darussalam.

6. Recommended Methods for the Identification and Anlysis of Amphetamine, Methamphetamine and their ring-substituted analogues in seized materials. (2006). *United Nations Office on Drugs and Crime.* New York.

7. Clarke, E. (2004). *Clarke's Analysis of Drugs and Poisions* (Third Edition ed., Vol. Volume 1 & 2). (A. Mofatt, M. Osselton, & B.Widdop, Eds.) London, Great Britain: Pharmaceutical Press.

8. *Pill Testing, Wikipedia*. (n.d.). Retrieved June 29, 2009, from Wikipedia: http://en.wikipedia.org/wiki/Pill_testing

9. Samuels, J. E. (2000, July). Color Test Reagents/Kits for Preliminary Identification of Drugs of Abuse. *National Institute of Justice*.

10. Pointer, D. S., & Ali, A. R. (2005). *Drug Testing Kits.* Australian Government, Department of Health and Ageing, South Australia.

11. O'Neal, Crouch, D., & Fatah, A. (2000). Validation of twelve chemical spot tests for the detection of drugs of abuse. *Forensic Science International* , 189-210.