

DrugNet Asia

Year 2008 Issue 7

A Regional Newsletter for Participating Laboratories at the UNDCP Consultative Meeting of Heads of Drug Testing Laboratories in Southeast Asia

Editor's notes

Very quietly, clandestine laboratories have been sprouting up in unassuming locations in the region and these laboratories have been discovered in the Philippines, Malaysia, Indonesia, and Cambodia. It is hence very timely that this issue is able to share with all readers the theme "Clandestine Laboratory and its Products" as encountered by the different drug testing laboratories. We thank all laboratories for their overwhelming responses to the call for articles and we hope that the information contained in this issue are relevant and useful to all. Happy reading!

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Clandestine Drug Manufacturing in the Philippines

Illicit drugs are traditionally smuggled into the Philippines. However, in 1997, a clandestine laboratory producing methamphetamine was discovered in Central Luzon. A theory was then raised suggesting that drug syndicates were considering the option of using the Philippines as a production site. Two years later, two other clandestine laboratories producing methamphetamine were also discovered and dismantled. The discovery of these two laboratories seemed to confirm the theory – the drug syndicate had shifted their strategy from plain trafficking to illicit drug production.

In addition, a new trend was recently observed in the clandestine laboratories' operations in the Philippines. There had been an increasing trend for the production process to be carried out in stages, with separate and distinct areas for each stage of production unlike the traditional method where the whole production process was done in one location. This method

of segregating the production process reduced the possibility of the various site being detected.

On 21 December 2006, the Philippine Drug Enforcement Agency (PDEA) together with the China National Narcotics Control Commission (CNNCC) busted a large scale, sophisticated methamphetamine clandestine laboratory at #343 McArthur Highway Barangay Caniogan, Calumpit, Bulacan.

Approximately 200 L of liquid methamphetamine hydrochloride, 3.6 kg of ephedrine, large amounts of precursors and essential chemicals and eight sets of sophisticated methamphetamine-producing equipment were seized.

Thionyl chloride, red phosphorous, palladium chloride and iodine were not recovered from this clandestine laboratory. However large volumes of acetone, ethanol, hydrochloric acid and caustic soda were found.

(continued on page 4)



Fig. 1: Clandestine Laboratory Facility



Fig. 2: Stirrer with reaction vessel



Fig. 5: Gas cylinder and pump



Fig. 6: Bags of ephedrine



Fig. 3: Liquid methamphetamine in metal basin



Fig. 4: Boxes of solvents



Fig. 7: Rotary evaporator



Fig. 8: Methamphetamine HCl taken from metal basin

Two officers from the Centre for Forensic Science, HSA, recently attended a one-week training course on Basic Clandestine Laboratory Safety in Drug Enforcement Administration (DEA) academy which is based in Quantico, Virginia, USA. This course is recommended for enforcement officers and chemists who are involved in the investigation of clandestine drug laboratories.

This was their account of the training course:

The training programme comprised both theory and practical components, with emphasis on methamphetamine (or "ice") production. The instructors were from Network Environmental Systems (NES) and DEA.

The topics covered include:

Methamphetamine Synthesis Method
Red Phosphorous method
Lithium/ammonia or Nazi method
Phenyl-2-propanone (P2P) method
Thionyl chloride method

❖ Different Methamphetamine Production Methods

We were shown how to synthesize methamphetamine using the Red Phosphorus method and Lithium/Ammonia method at the Mid-Atlantic Laboratory in Maryland.

❖ Field Hazards

We were introduced to the National Institute for Occupational Safety and Health (NIOSH) pocket guide to Chemical Hazards. This pocket guide (www.cdc.gov/niosh) contains the following information for 677 common chemicals:

- ♦ Chemical names and formulas
- ♦ Exposure limits – Immediately Dangerous to Life and Health (IDLH) values, NIOSH recommended exposure limits (RELs), OSHA permissible exposure limits (PELs) and the recommended measurement methods
- ♦ Chemical and physical properties, incompatibilities and reactivities
- ♦ Personal Protection / Sanitation
- ♦ Respirator recommendations
- ♦ The exposure routes, symptoms of exposure as well as target organs
- ♦ Procedures for emergency treatment (First Aid)

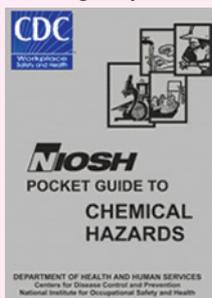


Fig. 1: NIOSH Pocket Guide to Chemical Hazards

❖ Field Hazards – Physical and Chemical Hazards

Some of the common physical hazards encountered in a clandestine laboratory include improper container handling,

heat stress, cold stress, electrocution, fatigue, fall, radiation, poor visibility and confined spaces. Heat stress and cold stress are often aggravated by protective clothing.

Chemical hazards arise from the presence of chemicals in a clandestine laboratory. The knowledge of chemical properties is therefore important as it helps to anticipate the potential hazards that one may encounter.

❖ Hazard Recognition - Air Monitoring

In order to assess the quality of air inside a clandestine laboratory, we should be armed with devices such as a combustible gas indicator (CGI) and drager pump (Fig. 2). For this session, we learned how to use these devices and interpret the results for field applications. These devices have to be calibrated regularly to ensure their accuracy and precision.



Fig. 2: Combustible Gas Indicator (top left), Drager Pump (top right) and Drager Detection Tubes (bottom right and left)

❖ Hazard Protection - Hazard Control

Hazard control refers to the methods and procedures used to reduce or prevent exposure to chemical, physical and biological hazards. There are three main hazard control methods, namely engineering, administrative and personal protective equipment.

Level	Items	Recommended Use	
D	Work clothes Coveralls Leather boots Leather gloves Hard hat Safety glasses	Not for chemical hazard For minimal physical hazard	<div style="display: flex; align-items: center;"> <div style="writing-mode: vertical-rl; transform: rotate(180deg); color: red; margin-right: 5px;">Level of Hazard Threat</div> <div style="writing-mode: vertical-rl; transform: rotate(180deg); color: green; margin-right: 5px;">Level of Protection</div> </div>
Modified D	Chemical suit (Tyvek) Boots Gloves Eye Protection No respirator	For minimal chemical contact Used in environmental work	
C	Chemical suit (Tychem SL) Boots and booties Inner/outer gloves Air Purifying Respirator (APR) Tape-up	For moderate skin hazard For moderate inhalation hazard For laboratory processing (select after laboratory assessment) For minimal atmospheric hazard	
B	Chemical suit (Tychem SL) Boots and booties Inner/outer gloves Self-Contained Breathing Apparatus (SCBA) Tape-up	For high skin hazard For high inhalation hazard For laboratory assessment Safety stand-by Unknown atmosphere	
Modified B	Level B suit-up (inner) Partially encapsulating suit Not gas tight	For very high skin hazard For very high inhalation hazard For hazardous atmosphere Examples: Fentanyl, LSD, PCP	
A	Work uniform Inner/outer gloves Self-Contained Breathing Apparatus (SCBA) Fully encapsulating suit Gas tight	For highest skin hazard For highest respiratory hazard For highly hazardous atmosphere For highest hazardous atmosphere Examples: Fentanyl, LSD, PCP	

Table 1: Different levels of personal protection for processing a clandestine laboratory

Laboratory Investigation

Engineering controls include ventilating the location, eliminating the source of ignition and removing the chemical sources.

Administrative methods help to reduce duration of exposure. They are always implemented regardless of the situation. Some examples of administrative controls include adding or rotating team members to complete the work faster, reducing heat stress by working at night, using safety equipment, following proper decontamination procedures and minimizing exposure by maintaining distance.

Personal protective equipment serves to isolate the body from the hazards. The effectiveness of this method is highly dependent on the proper selection and usage of the equipment. Table 1 shows a system that is used to assist the personnel in the selection of appropriate protective equipment based on the various degrees of hazard threats encountered.

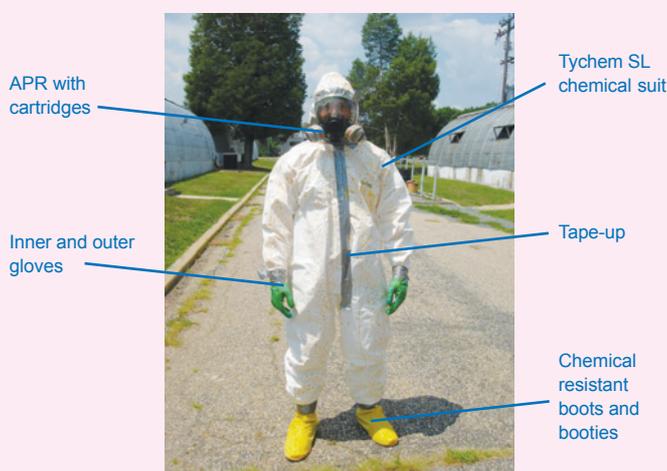


Fig. 3: Example of a Level C protection

❖ Field Safety Operations - Site Control

Site control serves to prevent and reduce unwanted spread of contamination via field procedures that are simple but critical. Each site can be divided into three work zones, which are as follows:

- Exclusion zone – Area of known or suspected hazards
- Decon zone – Access between exclusion and support zone
- Support zone – Area which is free from hazards

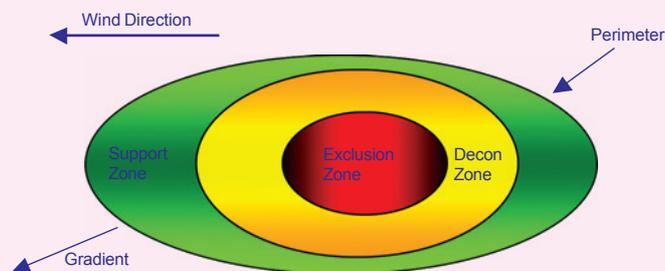


Fig. 4: Typical work zones of a site

❖ Field Safety Operations - Decontamination Set up

Decontamination must be carried out in the decon zone. A combination of chemical decon (wet) and physical decon (dry) can be used. Fig. 5 shows a typical decon set up.

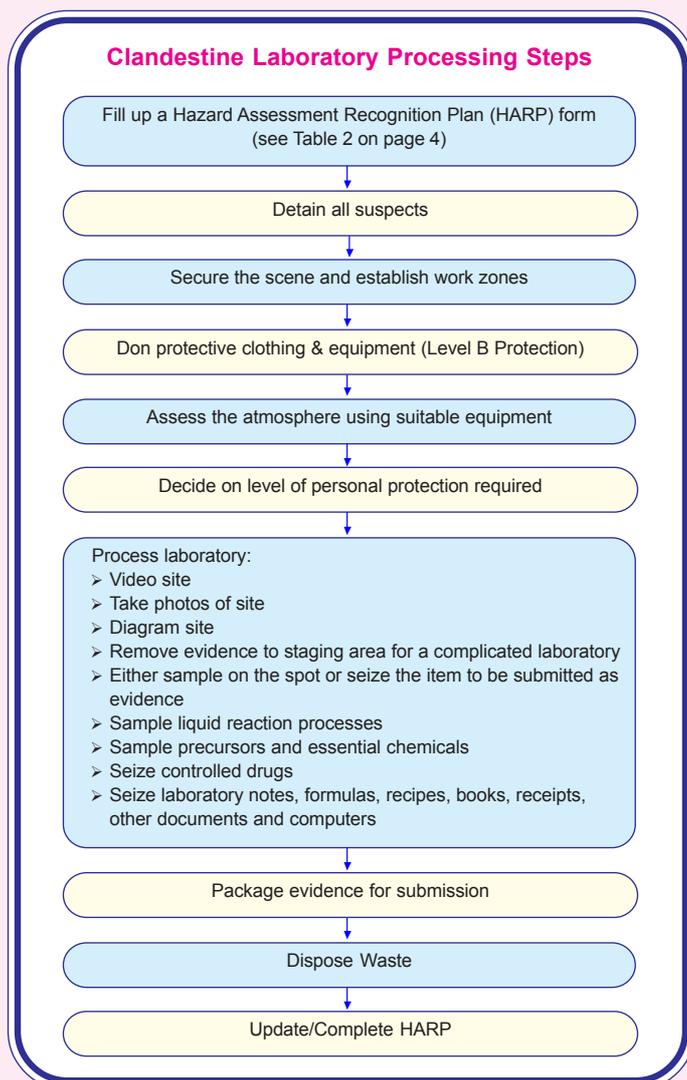


Fig. 5: Typical decon set up

❖ Hazardous Waste Cleanup

The disposal of items seized in a clandestine laboratory can be the responsibility of either the law enforcement department or the laboratory depending on regulations.

A useful reference guide for Hazardous Waste Cleanup is the “Guidelines for the Safe Handling and Disposal of Chemicals used in the Illicit Manufacture of Drugs” by United Nations Office on Drugs and Crime (UNODC). This guide outlines the different approaches to tackle disposal issues in different circumstances.



~Contributed by the Centre for Forensic Science, HSA, SINGAPORE~

“Ecstasy” Tablets Encountered in Hong Kong

“Ecstasy”, commonly referred to 3,4-methylenedioxymethamphetamine (MDMA), is one of the most popular abused illicit drugs encountered in rave parties in Hong Kong. However, “Ecstasy” is now currently used more generally and the seizure may not contain MDMA only but rather a mixture of MDMA and a combination of some other drugs such as methamphetamine, ketamine, 4-bromo-2,5-dimethoxyphenethylamine (2C-B), methylenedioxyamphetamine (MDA) or caffeine. In some cases, MDMA may not be present in the “Ecstasy” tablets.

In Hong Kong, seized “Ecstasy” specimens were mostly in the form of round coloured tablet with a comical or brand name logo embossed on one of the surfaces (Fig. 1). Not only being different in colour, some tablets were different in physical form such as in the shape of square, triangle, heart and octagon (Fig. 2). The laboratory has recorded more than 600 different types of “Ecstasy” tablets since 2001.



Fig. 1: “Ecstasy” tablets recently seized in Hong Kong



Fig. 2: “Ecstasy” tablets in different shapes

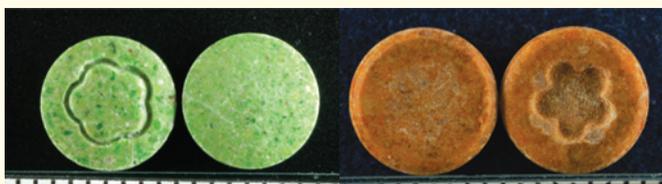


Fig. 3:(a) (left) Tablet with low MDMA content mixed with ephedrine, caffeine and methorphan. Diameter: 8 mm, Weight: 268 mg.
(b) (right) Tablet with high MDMA content mixed with methamphetamine. Diameter: 8.5 mm, Weight: 327 mg



Contents: MDMA Contents: MDMA, MA, 2C-B Contents: MDMA, MA, 2C-B Contents: MDMA, MA, MDA, DMA

Fig. 4: Tablets with the same “mushroom” logo but in different colours

The typical weight of “Ecstasy” tablet seized in Hong Kong was about 200 mg with an average MDMA content of about 70-100 mg per tablet. However, the laboratory had also encountered some tablets of atypical strength. The MDMA content might be as low as 2 mg (Fig. 3a) or as high as 190 mg per tablet (Fig. 3b). It is interesting to note that some of the examined tablets shared the same logo imprint, but they differ in colours and their compositions might vary (Fig. 4-6). In some of the seizures, highly similar tablets could have very different compositions (Fig. 7).



Contents: MDMA: 104 mg/tablet Contents: Ephedrine, 2C-B



Contents: MDMA: 108 mg/tablet Contents: MDMA: 109 mg/tablet, K, MDPP

Fig. 5: Tablets with the same “Disney” logo but in different colours



Contents: MDMA: 65 mg/tablet, MA: 3 mg/tablet, 2C-B Contents: MDMA: 33 mg/tablet, MDA: 32 mg/tablet, MA: 2 mg/tablet



Contents: MDMA: 54 mg/tablet, MA: 3 mg/tablet, 2C-B Contents: MDMA: 33 mg/tablet, MDA: 33 mg/tablet, MA: 1 mg/tablet

Fig. 6: Tablets with the same “CS” logo but in different colours



Contents: MDMA: 50 mg/tablet, MDA, MA: 3 mg/tablet, DMA, Caffeine, K Contents: MDMA: 45 mg/tablet, MA: 2 mg/tablet, 2C-B



Contents: MDA: 112 mg/tablet, K: 2 mg/tablet Contents: Ephedrine, 2C-B

Fig.7: Tablets with similar logos and colours but with different compositions

~Contributed by the Government Laboratory, HONG KONG SAR~

First Clandestine Methamphetamine

In April 2007, for the first time in the country, a large clandestine methamphetamine laboratory was discovered and dismantled in Cambodia. It was located in a cattle ranch at Kompong Speu, about 80 km south west of Phnom Penh. Significant quantities of precursor chemicals and some processing equipment were confiscated. Seventeen suspects were arrested, including four foreign nationals.



Fig. 1: Front view of clandestine laboratory



Fig. 2: Back view of clandestine laboratory

Summary of Findings at the Clandestine Laboratory

Chloroephedrine was produced by reacting pseudoephedrine hydrochloride with thionyl chloride. Among the precursor chemicals seized were:

- About 3.2 tons of thionyl chloride packed in over seven hundred 2.5-litre bottles and 14 jerry cans
- Four 200-litre drums of acetone
- About 50 plastic buckets with various quantity of chloroephedrine/ether
- About 52 kg of purified chloroephedrine

Numerous empty 500 ml bottles (with their original ether and chloroform labels) were found in boxes stacked in the open ground. NACD forensic laboratory identified the chloroephedrine by colour tests (blue coloration with Simon's) and GC/FID. Using a portable FTIR with ATR, DEA chemists determined that several empty plastic bags found at the scene had traces of pseudoephedrine hydrochloride. This was subsequently found to be (+)-pseudoephedrine hydrochloride

by HPLC (ODS and chiral column) by the Department of Chemistry, Malaysia.

Another 700 kg of potassium hydroxide and several sacks of activated charcoal were uncovered from a Phnom Penh warehouse suspected to be linked to this clandestine laboratory.

Based on the evidence at the site, it was concluded that this clandestine laboratory was only doing the first stage of the Emde process where the intermediate chloroephedrine was produced from (+)-pseudoephedrine hydrochloride. There was no evidence of conversion of the chloroephedrine to methamphetamine at this site.

Cleanup of the Clandestine Laboratory

GSM Consultancy, a specialist waste disposal company based in Malaysia, was contracted by UNODC to destroy the extremely hazardous thionyl chloride. The disposal exercise which was funded by the US and Australian governments afforded the Cambodian staff from the various government agencies valuable hands-on training. About 20 local staff were trained on the proper use of personal protective equipment and how to perform the thionyl chloride neutralization in a safe and environmentally-friendly manner. Using the neutralization process (see box on page 7) developed by the waste disposal company, the local staff took 11 days to destroy the 3.2 tons of thionyl chloride.

Another 800 litres of acetone and over 100 kg of the chloroephedrine/ether mixtures were disposed by open burning (Fig. 21) while 700 kg of potassium hydroxide were neutralized with concentrated hydrochloric acid (Fig. 22) and disposed in a remote area. With the acquired training, skills and confidence developed over the two weeks, local staff would be able to use safe and efficient procedures to dispose chemicals and wastes from clandestine methamphetamine laboratories dismantled in the future.



Fig. 3: Boxes of thionyl chloride in 2.5 litre bottles



Fig. 4: Jerry cans of thionyl chloride, 20 kg each



Fig. 7: Chloroephedrine production area



Fig. 8: Close up view of the crude chloroephedrine



Fig. 5: Thionyl chloride (4.1 kg each)



Fig. 6: Thionyl chloride label



Fig. 9: Crude chloroephedrine in Buchner funnel



Fig. 10: Purified chloroephedrine

Amphetamine Laboratory in Cambodia



Fig. 11: 10 tons of NaHCO₃



Fig. 12: Working out the optimal mixture



Fig. 13: Morning safety briefing



Fig. 14: Getting ready



Fig. 15: Suiting up



Fig. 16: Fit test for face mask



Fig. 17: Preparing the NaHCO₃/CaO mixture in a tub



Fig. 18: Pouring the thionyl chloride and hoeing



Fig. 19: Pouring thionyl chloride



Fig. 20: Adding H₂O₂ to the neutralized thionyl chloride



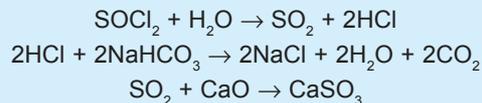
Fig. 21: Acetone burning in 55-gallon drums



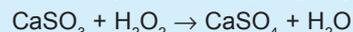
Fig. 22: Neutralization of KOH with conc. HCl

Thionyl Chloride Neutralisation

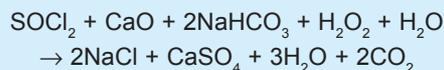
Thionyl chloride was neutralised by reacting with an excess mixture of sodium bicarbonate/calcium oxide in a tub. Each tub of neutralization mixture contained 300 kg of sodium bicarbonate and 100 kg of calcium oxide. A maximum of 30 bottles of thionyl chloride (123 kg) was destroyed per tub.



After neutralization, the calcium sulphite was oxidized to calcium sulphate with hydrogen peroxide.



Thus, the following reaction scheme describes the the whole neutralization process.



About 10 tons of sodium bicarbonate and 3.2 tons of calcium oxide were used in the neutralization process. Another 1 ton of 50 % hydrogen peroxide was used to oxidize the calcium sulphite to calcium sulphate.

~Contributed by the Laboratory of the National Authority for Combating Drugs (NACD), Phnom Penh, CAMBODIA~

Benzylpiperazine in Indonesia

Designer drugs of the benzyl- or phenyl-piperazine type have appeared throughout the world and are commercially available from the internet since January 2002. These designer drugs have been known to produce amphetamine-like effects.

Recently, benzyl piperazine drugs have appeared on the drug scene in Indonesia. Since November 2006, this laboratory has received several cases involving tablets, capsules and powders containing 1-benzylpiperazine. The weight of such tablets ranged from 500 – 600 mg.

The photos below show two types of benzylpiperazine tablets in their respective packaging. The tablets are suspected to originate from New Zealand.



Fig. 1: "Flying Star"



Fig. 2: "Red Dragon"

~Contributed by the National Narcotics Board Laboratory, Jakarta, INDONESIA~

Clandestine Methamphetamine Laboratory Using H₃PO₂ Route

In May 2007, a methamphetamine clandestine laboratory ran by four Nigerians was uncovered in Malaysia. Various equipment/paraphernalia for methamphetamine production, about 302 g of crystalline (+)-methamphetamine hydrochloride (78% as base), 50% hypophosphorous acid, iodine, 460 g of sodium hypophosphite, methylated spirits, sodium hydroxide and concentrated hydrochloric acid were recovered from the crime scene (Fig. 1 and Fig. 2). From a domestic food blender, traces of (+)-pseudoephedrine were identified by GC/MS and HPLC (ODS and chiral column). Sodium hypophosphite was identified by FTIR and SEM.

This is the first report of a clandestine methamphetamine laboratory utilizing the hypophosphorous acid/iodine synthetic route in the SE Asian region although this route had been commonly encountered in Australia since 1995.

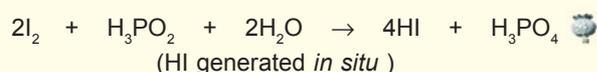


Fig. 1: Reflux setup with gas trap



Fig. 2: 50% H₃PO₂ in 500 ml bottles

~Contributed by the Department of Chemistry, MALAYSIA~

Project H44: Study Tour for Laboratory Analysts

A study tour was conducted for a laboratory analyst from each of the following countries: Cambodia, China, India, Indonesia, Lao PDR, Myanmar, Philippines, Thailand and Vietnam. It was held at the narcotics laboratory of the Forensic Division, Department of Chemistry, Malaysia, from June 18-19 2007. The two-week course included:

- Quantitative determination of ketamine, nimetazepam, methamphetamine and MDMA
- ATS clandestine laboratory investigations – sharing experiences; preparation
- Authentication and certification of secondary reference drug standards
- Moot court
- “Ecstasy” tablets identification database

Dr. Makino gave a two-day training session on the basics of methamphetamine profiling, differentiation of

ephedrine from pseudoephedrine and enantiomeric determination of methamphetamine, ephedrine and pseudoephedrine by chiral column HPLC.



Fig. 1: Dr Makino and participants at the Department of Chemistry Malaysia

~Contributed by the Project H44, UNODC, Bangkok~

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