Development of a Wipe Analysis Test Kit of Methamphetamine Residue by Gas-Chromatography – Mass Spectroscopy

Introduction

Over the last fifteen years, methamphetamine abuse has become a global problem [1]. The rising popularity of drugs, such as Ecstasy, GHB, and other methamphetamines has caused an increase in the number of clandestine drug laboratories in the United States and Europe [4]. Methamphetamine manufacturing accounts for 80-90 percent of the total drug production from clandestine labs [10]. This influx may be due to the evolution of simpler manufacturing procedures [4]. Previously, highly explosive, flammable, and toxic industrial chemicals were used as the precursor ingredients [4]. Now, with simpler “cold cook” methods, common household acids, caustics, and solvents are used [4]. This growing problem presents a challenge to both national law enforcement and forensic scientists [1].

With stiffer national drug laws, more accurate and specific methods of identifying and analyzing amphetamine-type stimulants have become an issue [1]. In addition, the timely exchange of analysis between forensic laboratories and law enforcement officials is needed to reduce drug trafficking [1]. The United Nations Office on Drugs and Crime (UNODC) Laboratory and Scientific Section has established a manual of “recommended methods of testing for national drug testing laboratories [1].” The purpose of this manual is to “assist drug analysts in the selection of methods appropriate to the sample under examination, leaving room also for adaptation to the level of sophistication of different laboratories [1].” Most methods described are published in scientific literature, and have been used for a number of years in reputable laboratories....however, a
number of other published methods in the forensic science literature also produce acceptable results [1]. In order for an analytical approach to adequately identify a controlled drug, as dictated by the jurisdiction, a minimum of two uncorrelated parameters must be determined [1]. The use of gas chromatography – mass spectrometry (GC – MS), a hyphenated technique, provides information from two parameters: retention time and mass spectrum [1].

For the purpose of this study, methamphetamine will be the amphetamine-type stimulant under investigation. The goal of this project is to develop a method using the GC – MS to detect the presence of methamphetamine residue and quantify it. In addition, a kit will be put together using the most adequate method for extracting methamphetamine residue from various surfaces, including linoleum, wood, granite, ceramic, metal, and sheetrock. There are several kits available for consumers to purchase to identify the presence of methamphetamine. These kits use different solvents, such as methanol and isopropyl alcohol, to extract methamphetamine. These solvents, along with others, will be analyzed at their ability to extract methamphetamine from different types of surfaces that are typically contaminated from a “cook”. The area needed for the wipe analysis will also be investigated. Most kits require a 4" x 4" area (100 cm²); others require a 6" x 6" area.

Literature Review

Amphetamine-type stimulants are a group of substances, derived structurally from β-phenethylamine, which stimulate the central nervous system, posing a risk of psychotic toxicity from overdose or long-term abuse [1]. Using these stimulants causes an increase in alertness, heart rate, blood pressure, respiration, body temperature and
euphoria [1]. Long-term abuse of amphetamine-type stimulants can result in agitation, tremors, hypertension, memory loss, hallucinations, psychotic episodes, paranoid delusions, and violent behavior [1].

International, national, regional, state, and local authorities are strategizing ways to deal with the illegal drug manufacturing, trafficking, abuse, and associating crimes, including clandestine methamphetamine labs [10]. Clandestine methamphetamine labs cause physical injury from explosions, fires, chemical burns, and toxic fumes, environmental hazards, and child endangerment [10]. Mixing chemicals creates substantial risks of explosion, fires, chemical burns, and toxic fume inhalation [10]. At this time it is not fully known the long-term health risks associated to such exposures [10].

There is little published literature regarding adverse health effects from exposure to illicit drug laboratories, especially from living in such environments [2]. Clandestine laboratories pose the highest risk of chemical exposure to the "cook", the individual directly involved in the chemical synthesis of methamphetamine [3]. However, there is also a risk to their assistants, emergency responders, hazardous material cleanup crews, neighbors, and future property occupants [10]. The "cook", the individual who mixes the chemicals, has little, if any, chemistry background [10].

With a high school chemistry background, or less, these "cooks" take little safety precautions [10]. There is a high risk of explosion from heating chemicals; an explosion can be triggered indirectly from smoking, electrical switches, or from the friction generated by equipment [10]. Typically, there is poor ventilation, increasing the toxic
fume inhalation and the risk of explosions [10]. Good ventilation, however, puts others at risk, as the toxic fumes spread outside [10].

There are two main types of clandestine laboratories: “super” labs and small-scale labs [10]. The labs are classified by the amount of product obtained in a production cycle. A “super” lab is highly organized and capable of manufacturing ten or more pounds of methamphetamine within 24 hours [10]. However, a small-scale lab is only capable of manufacturing one to four ounces of methamphetamine per production cycle. These “mom and pop” labs, as they are often referred, produce enough methamphetamine to support self usage [10]. A third type of lab, known as a “dirt” lab, is a desperate attempt to extract the residual methamphetamine from the dirt where toxic waste was dumped from a “super” lab [10].

Each pound of manufactured methamphetamine produces approximately five to six pounds of hazardous waste [10]. This waste is commonly disposed of improperly; burned, dumped along the road or into streams or rivers, poured down the drain, placed in household or commercial trash, or stored on the property [10]. Dumping toxic waste into dumpsters puts sanitation workers at risk [10]. Toxic waste can be transferred from surfaces and equipment onto the body and clothing of those in contact with the lab, and can subsequently contaminate other locations [10]. More research is needed to understand this toxic dumping’s long-term environmental effects [10]. Residual contamination of the ground, water supplies, buildings, and furniture may last for years [10].

An important role in the interpretation of analytical results of drugs of abuse stems from the knowledge of illicit manufacturing routes [1]. These processes involve
handling strong acids, strong bases, flammable solvents, and toxic chemicals as well as drug intermediates [2]. Exposure to ephedrine or other stimulant intermediates may lead to toxicity [2]. Methamphetamine can be absorbed through the skin causing rapid heart rate, hypertension, irregular cardiac rhythm, and in extreme cases, death [2]. Individuals with long-term exposure exhibit self mutilation, grinding of teeth, and in some instances seizures and death [2].

Exposure to solvents can lead to immediate toxicity; this includes intoxication, coma, and eventually death [2]. Chronic exposure to high levels of the types of solvents used in drug laboratories can lead to chemical hepatitis, renal failure, neuropathy, and death [2]. Exposure to concentrated acids or bases can lead to severe chemical burns, gastrointestinal injury, or eye injuries [2].

Iodine exposure is rather unique in that exposure to relatively small amounts of iodine (200 mg) has been associated with fatalities in children [2]. Inhalation exposure to iodine fumes has lead to pulmonary edema and pneumonitis [2]. Finally chronic exposure to iodine can lead to thyroid gland dysfunction [2].

Mercury salt exposure has been associated with severe gastrointestinal injury [2]. Late manifestations of mercury exposure include a characteristic skin rash and central nervous system toxicity [2]. Contamination of enclosed environments such as a home with elemental mercury has been associated with multiple illnesses among occupants [2].

Methamphetamine is a simple molecule and the EI mass spectrum has a single peak at m/z 58. For a complex matrix, such as urine, this is not useful for structural identification.
Similar to amphetamine, methamphetamine is a stimulant with longer lasting clinical effects [3]. Most commonly, it is synthesized from ephedrine or pseudoephedrine, found in cold tablets [3]. First propagated in California, methamphetamine labs were operated by criminals and motorcycle gangs [3]. Established throughout the United States, methamphetamine labs can be found in houses, apartments, hotel rooms, trailers, vans, and storage units [3]. These are just some of the structures used for labs, which may be moved frequently to prevent detection [3]. Rural areas are preferred locations due to the privacy, but no area is immune to these growing clandestine labs [3].

The Washington State Department of Health has information on at least 59 residential structures that were declared unfit for use as a result of chemical exposure contamination by clandestine drug labs in 1996 [3]. Operating laboratories present the greatest exposure hazard from release of reagent chemicals and their byproducts and potential for fire or explosion [3]. Former labs, where all the reaction vessels have been removed, pose little acute exposure hazard but may still cause illnesses in subsequent inhabitants [3].

In the past, methamphetamine production involved the use of several highly flammable and toxic industrial chemicals including cyanide compounds, mercury, and ether [4]. Many of these substances were subsequently placed on a list of watched chemicals by the US Drug Enforcement Administration [4]. As a result, methamphetamine manufacturers developed alternative methods using common household chemicals [4]. Yields are low, but these methods are popular because the precursor chemicals are accessible in communities in the United States [4]. One of the
most popular method is the ephedrine reduction method [4]. Ephedrine is separated from other ingredients in nutritional supplements, cold medicines, and herbal pills by boiling pills in hydroiodic acid and red phosphorous [4]. Iodine is obtained from pet stores, and the red phosphorous is obtained from match book striking pads [4]. After boiling, the iodine and red phosphorous are filtered out, and the ephedrine is converted to methamphetamine using sodium hydroxide (drain opener lye), hydrochloric acid or hydrogen sulfide, and a hydrocarbon solvent (toluene or camping stove fuel) [4].

Byproducts are poured down the drains of apartments and homes where the laboratories are located, resulting in serious chemical and thermal burn hazards for law enforcement officers, maintenance and cleanup personnel, and future residents [4].

As sale restrictions make it difficult to obtain precursor chemicals, the recipes for manufacturing methamphetamine continue to change [3]. These recipes can be obtained from publications, the Internet, or passed from person to person [3]. Due to the variability in the recipes, the exact hazards present in a methamphetamine lab is unpredictable [3]. Labs contain corrosives, solvents, drugs, and potentially other poisons [3]. Corrosives include strong acids and bases, often hydrochloric acid and sodium hydroxide [3]. Many types of solvent may be found in methamphetamine labs—ether, toluene, denatured alcohol, Freon, and more recently a type of gasoline used in camping stoves [3]. Methamphetamine, ephedrine, and pseudoephedrine are also common [3]. Other poisons include mercury and lead [3]. The laboratories also many contain explosives or toxic "booby-traps [3]." Thus, only specially trained investigators should enter methamphetamine labs [3]. Exposure to corrosive substances may produce symptoms ranging from shortness of breath and cough or chest pain to skin
burns [3]. Many solvents are absorbed into the body through inhalation and dermal exposure, and may cause dizziness, disorientation, headache, and nausea [3]. Our study of law enforcement personnel involved in the investigation of methamphetamine labs indicated that their symptoms were primarily headache and respiratory, mucous membrane, and skin irritation [3]. Lead-contaminated methamphetamine has caused lead poisoning in methamphetamine users, but lead poisoning has not otherwise been reported from exposure to methamphetamine labs [3]. Children living in methamphetamine labs are at risk for inhalation, transdermal skin absorption, and ingesting chemicals [3]. Children exposed to methamphetamine labs may develop respiratory symptoms, mucous membrane irritation, nausea, and headache [3]. Adverse health effects have been reported in subsequent occupants of labs that have not been adequately cleaned up [3]. Examples include throat irritation, respiratory difficulties, and headache in families who unknowingly moved into houses that previously contained methamphetamine labs [3]. Residual chemicals such as mercury, lead, methamphetamine, and caustic substances may pose health concerns in residential structures even after the laboratory equipment has been removed [3]. Areas of skin exposed to hazardous chemical liquids and solids should be thoroughly washed with soap and water [3]. Persons performing decontamination should wear suitable protection, such as chemically protective clothing, gloves, and respiratory protection when appropriate [3]. Minimal exposures such as walking near drug lab do not require decontamination [3]. The public health risks from potential chemical contamination in methamphetamine labs vary tremendously from site to site with differences in chemicals used, quantity of manufacture, extent of ventilation, and other factors [3]. Prior to
reoccupation, the structure must be cleaned [3]. The process involves initial
determination of the extent of contamination, followed by thorough cleaning, primarily
with detergent and water, and retesting to determine the adequacy of cleanup [3].
Typical cleanup costs range from $3500 to $5000, but in certain cases may exceed
20000 [3]. Reoccupation testing used in Washington includes wipe samples for pH and
methamphetamine levels, and air sampling for volatile organic compounds, and lead
and mercury when indicated [3].

Injuries from corrosive and caustic chemicals are increasing due to the shift to
"cold cook" methods for manufacturing methamphetamine [4]. These laboratories pose
health hazards for police, firefighters, and the public [4]. Illicit drug use in the United
States reached a high of 12.9 million persons in 1979, decreased through the 1980s to
a low of 12 million in 1992, and increased in the late 1990s to 13.6 million [4]. The
increase is due largely to the popularity of four clandestine drugs: methamphetamine,
MDMA, Rohypnol, and GHB [4]. The advent of simple methamphetamine production
methods has allowed clandestine drug manufacturers to circumvent diversion controls,
and has resulted in a dramatic increase in the number of clandestine laboratories in the
United States [4]. In addition, the Internet has allowed clandestine methamphetamine
manufacturers in Europe and North America to exchange information on production
methods [4]. Methamphetamines can be smoked, inhaled, injected, or ingested;
resulting in increased heart rate, blood pressure, body temperature, and rate of
breathing [4]. Side effects include increased violent behavior, nervousness, irritability,
paranoia, and severe depression upon withdrawal [4].

Methodology
Items to investigate to create a kit to identify methamphetamine: Find a qualitative method to identify methamphetamine accurately and efficiently on the GC-MS. Qualitatively identify methamphetamine in samples. Quantitatively identify concentration of methamphetamine in samples. Is an internal standard needed to quantify methamphetamine? Find an internal standard to use with methamphetamine. Find a solvent that effectively extracts methamphetamine from surfaces quantitatively. Solvents to be tested: acetone, ethanol, ether, hexane, isopropanol, methanol, toluene, and trichloroethane. These solvents are used to extract ephedrine from cold tablets; therefore, they should be able extract methamphetamine. Damage to surfaces may be a factor in determining a solvent. Find a method to extract methamphetamine from wipe samples. Determine the material needed to do wipe analysis — alcohol prep (cotton soaked in 70% isopropyl alcohol, 2" x 2" cotton gauze, or Whatman 40 filter paper. Test wipe analysis on different surfaces commonly found in a home, to determine if method works, realistically. Surfaces to be analyzed are plain sheetrock, glossy painted sheetrock, matte painted sheetrock, wallpapered sheetrock, painted wood trim, unpainted wood, varnished wood, particle board, linoleum, Formica, glass, metal, and plastic. Quantify results from various surfaces. Test area needed to for wipe analysis: 4" x 4" area (100 cm²), 6" x 6" area, or smaller. Go out to sites and test methamphetamine kit, extract samples and analyze samples on GC-MS, qualitatively and quantitatively.

Extraction method: Take cotton gauze and soak it in solvent. Remove excess solvent, so the gauze isn’t dripping. Wipe surface in a "Z" pattern using the soaked gauze. Fold in half and wipe in a "N" pattern across surface. Fold in half again and
place inside a 10 mL plastic syringe. Using a plastic weighing dish, add 2 mL of HPLC grade methanol. Plunge the syringe, so the gauze is compressed at the bottom and uptake the 2 mL of methanol. With a gloved hand, cover the tip of the syringe and shake methanol through the gauze. Plunge the solvent into the weighing dish. Uptake the solvent, shake the syringe, and plunge again for a total of three times. Using a disposable pipet, place the extract into a sample vial for the GC-MS and run the samples.