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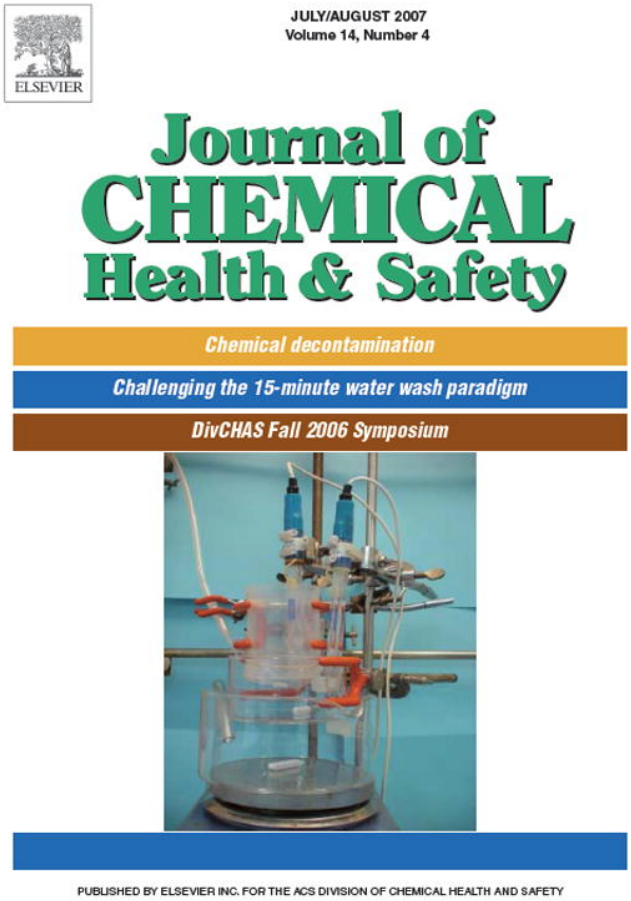

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FEATURE

Chemical concentrations and contamination associated with clandestine methamphetamine laboratories

We conducted a study to determine the chemical exposures associated with the clandestine manufacture of methamphetamine. Two scenarios were utilized, sampling at actual clandestine laboratories as they were being raided by law enforcement (Scenario 1) and sampling at controlled "cooks" conducted in houses to be destroyed (Scenario 2). Sampling during Scenario 1 revealed that most suspected laboratories had significant amounts of methamphetamine surface contamination throughout the suspected "cook" area. Levels of hydrocarbons, phosphine, iodine, and inorganic acids were unremarkable in these inactive laboratories. Sampling during the controlled cooks (Scenario 2) revealed high concentrations of phosphine, iodine, anhydrous ammonia, and hydrogen chloride during the "cooking" process. Anhydrous ammonia and hydrogen chloride were detected at levels that exceed NIOSH Immediately Dangerous to Life and Health (IDLH) levels. An aerosol of methamphetamine was also created during the process resulting in surface contamination within the structure as well as contamination on the clothing of the individuals participating in the "cooking" process. Based on our study, individuals entering a suspected clandestine methamphetamine laboratory should wear chemically resistant protective clothing and use a self-contained breathing apparatus. Individuals entering the suspected laboratory should also assume that items and persons associated with the "cook" area are chemically contaminated and need to be decontaminated.

By John W. Martyny, Shawn L. Arbuckle, Charles S. McCammon Jr., Eric J. Esswein, Nicola Erb, Mike Van Dyke

INTRODUCTION

The Nation continues to face an epidemic of clandestine methamphetamine drug manufacturing. Illegal laboratory seizures have increased nationwide from 7,438 in 1999 to 12,484 laboratories in 2005.[1] These clandestine labs continue to put police, fire, and other first responders at risk for a variety of hazards. In addition, susceptible third parties, such as children, are at risk for exposures to the chemical hazards as well as the fire, explosion, and safety hazards inherent with the clandestine manufacture of methamphetamine.

The Centers for Disease Control reported a number of public health injuries and illnesses in first responders and medical personnel associated with clandestine methamphetamine laboratories between 1996 and 1999.[2] One hundred and twelve methamphetamine-associated events were reported by five state health departments. These

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1871-5532/$32.00
doi:10.1016/j.jchas.2007.01.012

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events resulted in injury to 155 persons, of which 79 were first responders and 7 were hospital personnel. Predominant complaints in first responders were respiratory irritation and eye irritation, while hospital personnel complained primarily of nausea/vomiting and dizziness.

The State of Washington reported on 91 methamphetamine-related incidents, of which 35 (38%) resulted in injuries to a total of 66 people.[3] Twenty-two (33%) of the individuals injured were classified as members of the general public, but most were either methamphetamine "cookers" or individuals living in homes where methamphetamine was produced. Nineteen individuals were employees of businesses (hotels, refuse pickup, transfer facilities, etc.) where methamphetamine had been produced or byproducts were illegally dumped. Thirty-two (48%) of the 66 total people were hospitalized or taken to the hospital and released. The rest of the individuals were either treated at the scene, by their personal physician, or did not need treatment.

Studies conducted by Dr. Jefferey Burgess[4,5] investigated symptoms reported by emergency responders during illegal methamphetamine laboratory seizures. Responders predominately reported general irritant symptoms, but at least one case of phosphine gas exposure was reported. In the questionnaire study of emergency responders, 53.8% reported at least one illness while conducting laboratory seizures with most symptoms appearing to be related to chemical exposure at the laboratory site. The primary symptoms reported were headache and mucous membrane irritation.

Upon repeat pulmonary function testing, a number of responders were found to have an accelerated drop in 1 second forced expiratory volume (FEV_1) that may have been related to work in drug laboratories.[5] The majority of symptoms reported by officers occurred during the processing phase of the laboratory seizures which is also the phase in which the most time was spent in the laboratory area dismantling the laboratory and collecting evidence. The use of respiratory protection did seem to reduce the incidence of symptoms while investigating these laboratories. While there has also been anecdotal evidence of exposure to methamphetamine or methamphetamine laboratory byproducts causing permanent lung damage, actual cases have not been reported in the literature.

Due to these potential health effects, many law enforcement and social services agencies have developed policies for medical surveillance, personal protective equipment, and personal decontamination. These policies have been implemented based on limited evidence for chemical exposure in clandestine laboratory environments. This is the first systematic effort to assess potential chemical exposures associated with these environments and to provide recommendations based on quantitative chemical sampling results.

MANUFACTURING METHODS

Methamphetamine was first commercially synthesized in the 1930s and was used in many prescription and over-the-counter medicines until its long-term addictive effects were known. Prior to the 1990s, the clandestine production of methamphetamine was mostly confined to the Pacific coast states and was controlled by motorcycle gangs. The predominate early production method utilized phenyl-2-propanone (P2P) as the precursor. This manufacturing process can be very malodorous, difficult to conduct and requires some knowledge of chemistry. In addition, it produces a lower quality drug with less addictive properties as compared to the current production methods. In the 1988 the Federal Chemical Diversion and Trafficking Act of 1988 placed P2P and other chemicals on the controlled substances list, which increased the difficulty of obtaining the precursor chemicals for the P2P method.[6,7]

As P2P manufacturing method precursors became harder to obtain, clandestine chemists began to utilize production methods using ephedrine or pseudoephedrine as precursors in the production process. These compounds are structurally very similar to methamphetamine with ephedrine

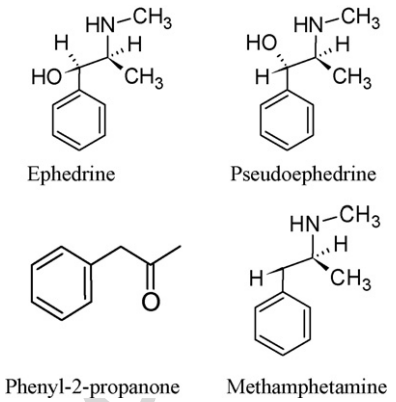

Figure 1. Illustration of the structural similarities between the ephedrine, pseudoephedrine, and methamphetamine. Phenyl-2-propanone was also utilized in the production of methamphetamine at one time.

differing only by a single hydroxyl group (Figure 1). In addition, this method of production yields a higher purity of d-methamphetamine, which is more physiologically active. Clandestine laboratories using the ephedrine/pseudoephedrine method of production are the most common laboratories found by law enforcement in recent years.[6–8]

The ephedrine/pseudoephedrine manufacturing processes have frequently been classified as three separate methodologies: the red phosphorous or "red P" method, the hypophosphorous acid method, and the Birch reduction method. The "red P" method and the hypophosphorous acid method are very similar with the only difference being the source of phosphorous used in the reaction. The Red "P" method typically uses red phosphorous, while the hypophosphorous method uses hypophosphorous acid. Both methods involve the addition of ephedrine/pseudoephedrine, iodine, water, and phosphorous in order to produce the methamphetamine. Both production methods utilize a strong base, a solvent, and hydrochloric acid to remove the methamphetamine from solution and both methodologies can produce large quantities of relatively high purity methamphetamine.[4,8]

The Birch reduction method has become very popular since the late 1990s due to the low cost and high

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availability of the necessary chemicals. This method combines ephedrine/pseudoephedrine with a reactive metal (sodium or lithium) in the presence of anhydrous ammonia. The need for a strong base for extraction is not necessary but the use of a solvent and hydrochloric acid is still necessary. Anhydrous ammonia is easily obtained, especially in rural areas where it is used as a fertilizer. Lithium is a reactive metal present within many photographic batteries. The nationwide incidence of clandestine methamphetamine production laboratories using this method rose from 439/3015 (14.5%) laboratories in 1998 to 2912/6426 (45.3%) laboratories in 2000, just a two-year period.[9]

As pure ephedrine and pseudoephedrine became more and more difficult to obtain, individuals interested in the clandestine manufacture of methamphetamine switched to extracting these compounds from cold tablets purchased or stolen from drug stores. This ephedrine/pseudoephedrine extraction method involves mixing the crushed pills with a light solvent (water or alcohol) in order to obtain the necessary compounds. The use of alcohol is faster since it evaporates faster, but it is much more flammable and can result in fires.[7]

We prioritized our exposure sampling based on information from law enforcement intelligence sources describing the precursor chemicals and anticipated production methods. Based on this information, the primary exposures of interest consisted of red phosphorous, hydrogen chloride, iodine, anhydrous ammonia, lithium, solvents, and sodium hydroxide. Sulfuric acid was also determined to be of interest when reacted with sodium chloride in order to produce hydrogen chloride gas. Heating of solutions containing red phosphorous has been shown to produce phosphine gas, an exposure concern due to its low threshold of toxicity. Lastly, methamphetamine aerosol was a concern since all manufacturing methods can release methamphetamine.[10]

STUDY OBJECTIVES

This study was undertaken to determine the potential chemical exposures to law enforcement, emergency services personnel, or individuals associated with the clandestine production of methamphetamine.

This study was undertaken to determine the potential chemical exposures to law enforcement, emergency services personnel, or individuals associated with the clandestine production of methamphetamine.

The goals of the study were to:

- Determine the primary chemical exposures of concern at clandestine drug laboratory seizures for first responders, children and adults present at the laboratory site.
- Determine magnitude and composition of the chemical exposures associated with the most common methods of manufacture.
- Determine which phase of the clandestine production process poses the greatest risk for responders and individuals associated with the manufacturing process.
- Determine the appropriate types of personal protective equipment required for the various phases of drug lab seizures based on exposure assessments.
- Determine the potential for chemical contamination of the structure used for the "cook" and subsequently the chemical contamination of individuals entering the structure during or after the "cook".

METHODOLOGY

Sampling Scenarios

Two types of sampling scenarios were conducted. The first scenario occurred during the investigation of suspected individual clandestine methamphetamine laboratories by law enforcement officers. In these situations, sample collection devices were brought into the suspected laboratory immediately after entry by law enforcement officials.

The second scenario involved controlled methamphetamine manufacture conducted in abandoned structures scheduled for destruction. This scenario simulated exposures during illegal methamphetamine manufacture. Samples were collected in the area of the "cooks" and at a distance from the "cook" in order to determine chemical exposures in different areas of the structure.

Laboratory Methods

Depending upon the method of methamphetamine manufacture, area air samples were collected for hydrocarbons, phosphine, inorganic acids, iodine, methamphetamine, and ammonia. In addition, surface wipe samples were collected for methamphetamine. All samples were analyzed by Data Chem Laboratories in Salt Lake City, UT.

During Scenario 1, sampling was performed for hydrocarbons, iodine, phosphine, inorganic acids, and metals (Al, Pb, P, Li, Mn, Fe, Zn, Ca, Cd) but as the investigation progressed, sampling for metals was discontinued because results were consistently below the limits of detections. Early in the study, samples were taken for organics using both summa canisters and thermal desorption tubes. After the first several labs, the use of the summa canister was eliminated since the thermal desorption tubes provided adequate information.

Later sampling focused on acids, iodine, and phosphine where illegal manufacturing appeared to have occurred in the recent past. Because surface wipe samples consistently showed positive results for methamphetamine, these samples were consistently collected in suspected laboratories.

Active sampling for hydrocarbons was performed using two different methods; vacuum canister collection and thermal desorption tube sampling. Stainless steel evacuated cylinders

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(summa canisters) were used to collect a known volume of air by taking the sampler into the lab and allowing the tank to fill. Samples were analyzed using gas chromatograph/mass spectrometer (GC/MS) according to United States Environmental Protection Agency (EPA) Method T0-15. The second method involved using Carbotrap thermal desorption tubes. Thermal desorption tubes consist of multi-layer charcoal sorbents through which a known volume of air is drawn using a flow-calibrated personal sampling pump. Samples were collected at a rate of 50 cubic centimeters (cc) per minute. After sampling, the tubes were sealed and packaged in air-tight containers and shipped to the laboratory for analysis using a GC/MS according to the EPA method T0-17.

Phosphine was sampled using mercuric cyanide treated silica gel tubes. Sampling trains were calibrated to a flow rate of 100 cc per minute. Samples were analysed according to NIOSH Manual of Analytical Methods (NMAM) 6002 using visible spectrophotometry.

Samples for inorganic acids, including hydrochloric acid were collected using a silica gel tubes with a sampling train calibrated to a flow rate of 200 cc per minute. Samples were analyzed according to NMAM Method 7903 using ion chromatography analysis.

Real-time sampling was also performed for HCl and phosphine using an ITX Multi-Gas Monitor (Industrial Scientific Corporation, Oakdale, PA) specific for these analytes.

Sampling for iodine was conducted using a sampling train calibrated to a flow rate of 1.0 Liters per minute (Lpm) with coconut shell charcoal tubes. Samples were analyzed using ion chromatography according to NMAM 600.

Samples for metals were collected using 37 mm sampling cassettes and 0.8 μm, mixed cellulose ester membrane filters. Sampling trains were calibrated to a flow of 2.0 Lpm. The filters were dissolved and analyzed using inductively coupled argon plasma spectroscopy according to NMAM 7300 for 27 elements.

Ammonia samples were collected according to NMAM 6015 (visible absorption spectrophotometry) using treated silica gel tubes. A sampling rate of 150 cc per minute was used for the sampling train.

Airborne methamphetamine samples were collected using 37 mm sampling cassettes and acid treated, glass fiber filters. The sampling train was calibrated to a flow rate of 2 Lpm. The samples were analyzed using a NIOSH method under development at the laboratory, which enabled the analysis of the samples using GC/MS.

Wipe samples for methamphetamine were collected by wiping hard surfaces suspected of contamination using a 4-in. by 4-in. (4 × 4) cotton gauze wipe. A single-use 100 cm^2 template was used if possible. Rarely, an irregular surface that did not lend itself to the use of the template (such as a hand) was sampled using an approximate 100 cm^2 area. Prior to entering the suspected laboratory, wipes were individually placed into plastic centrifuge tubes. After entering the laboratory, the wipes were taken out of the tubes and wetted with several milliliters of isopropanol or methanol prior to sampling. An attempt was made to minimize cross contamination by using separate pairs of gloves for each sample. After sampling, the wipes were put back into the centrifuge tubes and sent to Data Chem Laboratories for analysis. The samples were analyzed using a NIOSH method under development at the laboratory, which enabled the analysis of the samples using GC/MS.

Reference Levels

Chemical concentrations found during this study were referenced to the applicable Occupational Safety and Health Administration's (OSHA) Permissible Exposure Levels (PEL)[11] or to the American Conference of Governmental Industrial Hygiene's (ACGIH) Threshold Limit Values (TLV).[12] These levels are applicable to healthy workers and do not pertain to children or exposures to individuals outside of the occupational environment. They are reported in this study for comparison although they do apply to first responders.

In some instances, we have also compared observed concentrations to the NIOSH Immediately Dangerous to Life and Health (IDLH) levels.[13] These levels represent "conditions that pose an immediate threat to life or health, or conditions that pose an immediate threat of severe exposure to contaminants, such as radioactive materials, which are likely to have adverse cumulative or delayed effects on health." In determining IDLH values, "the ability of a worker to escape without loss of life or irreversible health effects" was also considered. Exposures to IDLH atmospheres should only be conducted with a high level of respiratory protection and unprotected individuals should leave or be removed from IDLH exposures immediately. Table 1 lists the available applicable comparison criteria for the measured chemicals.

RESULTS

Scenario 1

Fourteen suspected clandestine methamphetamine laboratories were

Table 1. Current occupational exposure levels for chemicals evaluated during this study

Chemical	OSHA PEL[11] (ppm)	ACGIH TLV[12] (ppm)	NIOSH REL[13] (ppm)	NIOSH IDLH[13] (ppm)
Ammonia	50	25	25	300
Hydrogen chloride	C 5	C 2	C 5	50
Iodine	C 0.1	C 0.1	C 0.1	2
Phosphine	0.3	0.3	0.3	50
Methamphetamine	None	None	None	None

ppm: parts per million; C: ceiling value which indicates that the exposure level may not be exceeded for any time period; none: no applicable occupational exposure standards have been promulgated by any of these organizations for methamphetamine.

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investigated during law enforcement operations. None of the laboratories were active at the time. In fact, most of the labs to which we responded were small, with limited amounts of chemicals and supplies. In only one instance had a "cook" occurred that day, according to law enforcement. We believe that these circumstances represent minimal levels of contamination and the exposures that we documented during the laboratory response phase are less than representative of exposures that might be expected at drug labs that are active (during a production operation).

Hydrocarbons

Results for hydrocarbons were difficult to interpret due to background concentrations of hydrocarbons from commercial products. Peaks were found for isopropanol, methanol, pentane, propene, toluene, heptane and a number of commonly used aliphatic hydrocarbons. Many common solvents are used in methamphetamine "cooks", so determining if these were unique when producing methamphetamine was not possible.

We did not observe any discrete concentrations of hydrocarbons except for isopropanol and methanol that were used as a solvent for our wipe samples.

Phosphine

Phosphine gas is liberated during the cooking phase of a phosphorous "cook". It is extremely reactive and not expected to be present unless a "cook" was occurring. We sampled for phosphine at three of the suspected laboratories with one laboratory result recording 258 ppm. However, a field blank was reported to contain 122 ppm, suggesting the method was biased, and that these results were likely false positives. Another field blank was reported as non-detectable, so it is possible that a systematic error occurred at the laboratory.

Inorganic Acids

Samples for acids were collected in six of fourteen laboratories. Samples were initially collected at all of the laboratories but were consistently less than the analytical limit of detection, so sampling for acids was discontinued.

Hydrogen Chloride

Hydrogen chloride was detected in only two of the clandestine labs sampled. In both cases, these were mobile homes. It is not clear that a laboratory had been recently in operation at either of these locations and the levels of acid found were low (0.005 ppm and 0.13 ppm). Detection of HCl could indicate that a "cook" had occurred within the suspected laboratory. These results may also represent the lower level of detection for this method. The current American Conference of Governmental Industrial Hygienists Threshold Limit Value for hydrogen chloride is a ceiling value of 2.0 ppm.[12]

Iodine

Samples for airborne iodine were taken at 10 of the suspected laboratories. In many of the laboratories, iodine stains were observed on carpeting and on the walls. It was expected, therefore, that iodine exposures might be high in some of these facilities. The results of the sampling are presented in Table 2.

Although iodine stains were readily apparent in many of the suspected laboratories, elevated levels of airborne iodine were not present in all of the locations. The levels of iodine that were found were low and well below the current ACGIH TLV of 0.1 ppm as a ceiling value.[12]

Table 2. Airborne iodine levels found during the survey of suspected clandestine methamphetamine laboratories during Scenario 1

Sample Location	Iodine (ppm)
Hotel room	ND
Upstairs closet	ND
Main room	ND
Upstairs bedroom	0.001
Main room	ND
Blank	ND
Hotel room	ND
Main room	0.002
Main room	0.0007
Upstairs	0.0008
Main room	ND
Downstairs	ND

ND: non-detect level (<0.0007 ppm); ppm: parts per million.

Methamphetamine Surface Wipes

Ninety-seven surface wipe samples were collected in the suspected methamphetamine laboratories (Table 3). Eighty-two samples (84%) were above the limit of detection and ranged from 1.0 μg/sample to 16,000 μg/sample. The 16,000 μg/sample was taken in a hotel room where there had been an explosion coating the ceiling with debris. The wipe was taken

Table 3. Results of methamphetamine wipe samples taken at suspected clandestine methamphetamine laboratories during Scenario 1

Lab Location Number	Number of Samples	Mean[a] (μg/Sample)	Median[a] (μg/Sample)	Range (μg/Sample)
1	5	134	120	6–370
2	5	202	28	9–920
3	6	150	26	1–150
4	6	3	2	ND–7
5	13	48	3	1–520
6	8	2788	925	71–16,000
7	5	3057	2400	25–10,000
8	7	42	37	13–64
9	9	96	17	1–430
10	7	312	250	64–790
11	9	46	1	ND–300
12	3	24	10	ND–63
13	3	438	650	4–660
14	3	33	14	8–78
Overall	89	511	28	ND–16,000

ND: none detected at levels above the limit of detection of 0.6 μg/sample.
[a] None detected samples were assigned a value of 0.01 μg/sample for the purpose of analysis.

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of the material on the ceiling. The overall arithmetic mean of all of the samples was 511 μg/sample. The median for all of the samples was 28 μg/sample. In eleven of the 14 suspected laboratories, all of the samples taken were positive.

Many of the locations where methamphetamine was found could not have been contaminated by material falling on a surface. Methamphetamine residue was found not only on tables, but also on air return grates and on ceiling fans. High levels of methamphetamine were found in refrigerators, microwaves, and kitchen appliances, suggesting that food contamination is likely to occur. In general, suspected clandestine methamphetamine laboratories had widespread levels of methamphetamine detected in many areas of the house or structure.

Figure 2. Controlled cook setup in a kitchen, used during Scenario 2 for sampling of emissions during a red phosphorous "cook". The apparatus used for the heating of the phosphorous, iodine, and ephedrine mixture is on the hotplate with the plastic tube going up and then down into a kitty litter container, typical of an actual "cook". Sampling devices are on the shelves above the kitchen counter.

Table 4. Controlled "cooks" conducted to determine exposures associated with clandestine methamphetamine production (Scenario 2)

Cook #	Structure Type	Manufacture Method Used	Runs Conducted[a]
1	House	Red phosphorous	1
2	Hotel	Red phosphorous	1
3	Duplex	Anhydrous ammonia	1
4	Duplex	Anhydrous ammonia	1
5	House	Anhydrous ammonia	1
6	House	Hypophosphorous	1
7	House	Phosphorous flakes	1
8	House	Red phosphorous	2

[a] The number of separate cooks that were conducted at that location.

Scenario 2

Controlled methamphetamine "cooks" were conducted in eight residential structures to determine potential concentrations of chemicals off-gassing from the manufacturing process. Concentrations measured during this scenario could however, represent exposures to an individual (or possibly a family) residing in the building where the manufacturing was conducted, as well as to first responders who might enter a suspected lab, during or shortly after an actual "cook". In all cases, the building was set up to use the chemicals, equipment and techniques representative of clandestine drug manufacturing activities (Figure 2). Current trends in the process were replicated to simulate the most realistic scenario possible. The structures sampled and the production methods utilized are presented in Table 4.

The chemicals and process used in the red phosphorous "cooks" and the hypophosphorous/phosphorous flake methodologies are similar. The chemicals sampled during the phosphorous methods were iodine, phosphine gas, hydrogen chloride, solvents, and methamphetamine. The production methodology used during the anhydrous ammonia "cooks" is much different than the phosphorous methods and requires different sampling methods. During the scenarios using the anhydrous methodology, we sampled for ammonia, hydrocarbons, hydrogen chloride, and methamphetamine.

Hydrocarbons

Consistent with results for Scenario 1, hydrocarbons associated with the "cook" could not definitively be ascribed to the illegal drug manufacturing process. Because white gas (camping stove fuel) was used as the solvent for the production phase, low levels of a large number of aliphatic hydrocarbons were detected. Methanol and/or isopropanol were notable and expected since they were used as solvents for the wipe samples.

Phosphine

Phosphine was detected in our first two red P "cooks" using NIOSH Method 6002 and using the Multi-Gas Monitor. There was a concern regarding the NIOSH method due to a previous high level in a blank. The results obtained during these first red phosphorous "cooks" were relatively low. In the "cook" area of the first "cook", the sample became overloaded with the result of 0.9 ppm phosphine. The level reported in the breathing zone of the "cook" was 0.1 ppm and the level at the remote area was 0.3 ppm. Samples taken at the second red phosphorous "cook" were all below the detection level for phosphine (<0.1 ppm). Peak concentrations of phosphine (obtained from the ITX Multi-Gas Monitor) were 0.55 and 0.84 ppm in the "cook" area. During the hypophosphorous "cooks", the direct reading monitor indicated 0.6 and 13 ppm in the "cook" area.

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Table 5. Ammonia levels obtained using NIOSH Method 6015 during controlled manufacture using the anhydrous ammonia method of methamphetamine production (Scenario 2)

Manufacturing Site	Cook Area TWA[a] (ppm)	Cook Breathing Zone TWA[a] (ppm)	Remote Area Sample TWA[a] (ppm)
Anhydrous #1	410	370	130
Anhydrous #2	190	130	<66
Anhydrous #3	338	310	366

ppm: parts per million.
[a] Time weighted average calculated by the laboratory as the average exposure over the entire sampling time.

Ammonia

During our initial anhydrous ammonia "cook" we used Multi-Gas Monitors to monitor ammonia levels. Within 5 minutes of initiating the "cook", the ITX Multi-Gas Monitor located in the "cook" area was over-range and within 16 minutes, all of the monitors in the building were over-range. The maximum level of ammonia recorded was 3348 parts per million (ppm). It is anticipated that this level was reached at the beginning of the "cook". The levels of anhydrous ammonia collected for laboratory analysis were also overloaded in two cases. The average ammonia concentration at the "cook" site during the first 3 hours of the "cook" was in excess of 410 ppm (Table 5). A significant amount of ammonia was found in the backup section of the tube indicating that the mean ammonia level at the "cook" area was higher than the calculated mean.

At the other side of the kitchen, the mean ammonia levels averaged 130 ppm for the same period of time and the tube was again found to have ammonia in the backup section indicating that more than 130 ppm was present. A personal lapel monitor worn by the DEA chemist indicated that the time weighted average ammonia level (for the first hour) of the "cook" was 370 ppm (Table 5).

Real-time samplers at the second anhydrous ammonia "cook" were also rapidly overloaded. Ammonia levels measured at the "cook" area averaged 190 ppm for the 2 hours of the "cook". The personal monitor that was placed in the breathing zone of the DEA chemist recorded a time weighted average concentration of 130 ppm, less than the level measured on the "cook" at the first location. This reduction in exposure was attributed to an evacuation fan that was installed in the window over the "cook" area.

After the second "cook", we used to Drager colorimetric tubes to measure the ammonia level without overloading a real-time instrument. The concentrations of ammonia measured using these tubes ranged from 500 ppm to 2000 ppm. In the "cook" area during the stirring of the mixture, the concentrations were generally at 2000 ppm. The concentrations gradually declined in the "cook" area if the solution was not agitated to a low of 500 ppm. Concentrations of 500 ppm were observed in the other rooms. The outside concentration at the window was about 50 ppm and the level upwind by the front door was measured to be 4 ppm. The anhydrous ammonia measured at the "cook" area was 338 ppm during the 101 minutes of the "cook" and 141 ppm during the 46-minute acidification phase. The levels across the room were 366 ppm during the "cook" and 268 ppm during the 46-minute acidification phase. The chemist conducting "cook" was exposed to a mean of 310 ppm even though he was only in the "cook" area for about 50% of the sampling period.

Hydrogen Chloride

Hydrogen chloride is used in all methods of production to precipitate the methamphetamine from the organic solvent as methamphetamine chloride. The results of the samples taken during the total "cook" phase suggest average concentrations during the entire manufacturing process while the samples taken during the extraction phase are

Table 6. Hydrogen chloride levels measured during controlled cooks to determine potential exposures during clandestine methamphetamine manufacture (Scenario 2)

Manufacturing Method	Phase of Manufacture	Cook Area (ppm)	Breathing Zone Sample (ppm)	Remote Area (ppm)
Red phos. #1	Total cook	9.61	0.43	0.11
Red phos. #2	Total cook	0.283	0.05	0.15
Red phos. #3	Total cook	0.27	NA	0.03
Red phos. #4	Total cook	0.42	NA	0.16
Hypophos. #1	Total cook	0.13	0.08	0.02
Hypophos. #2	Total cook	0.13	NA	0.21
Mean		1.81	0.18	0.11
Median		0.28	0.21	0.12
Red phos. #1	Extraction	20.0	6.12	0.79
Red phos. #2	Extraction	1.18	0.21	0.99
Hypophos. #1	Extraction	2.6	0.61	0.26
Hypophos. #2	Extraction	3.1	NA	4.5
Anhydrous #1[a]	Extraction	0.02	0.01	0.03
Anhydrous #2[a]	Extraction	0.02	0.1	0.03
Anhydrous #3[a]	Extraction	0.6	0.7	0.4
Mean		3.93 (6.72)	1.29 (2.31)	1.0 (1.63)
Median		1.18 (2.85)	0.41 (0.61)	0.6 (0.89)
Total mean[b]		2.95	0.92	0.59
Total median[b]		0.42	0.21	0.16

NA: not available; ( ): level of hydrogen chloride excluding the levels measured during the anhydrous ammonia methodology; ppm: parts per million.
[a] Interference noted by analytic laboratory.
[b] Mean and median calculated for both the total cook period of time and the extraction phase.

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those levels obtained just during the precipitation phase, a process that usually lasts for less than 1 hour. Exposures during the extraction phase are generally higher than during the total "cook" phase since there is very little hydrogen chloride present during the other parts of the manufacturing process. The average levels shown for the extraction phase could be much higher if not for an unknown chemical interference reported by the analytic laboratory during the analysis of the samples. Table 6 lists the results that were obtained from all of the "cooks".

Hydrogen chloride levels in the "cook" area during the extraction phase ranged from a low of 0.02 ppm during the anhydrous "cooks" to a high of 20 ppm during the red phosphorous "cooks". The current ACGIH TLV is a ceiling value of 2.0 ppm, indicating that hydrogen chloride levels may exceed current occupational exposure levels by as much as 10 times during the extraction phase.[12] Real-time measurements of hydrogen chloride using an ITX Multi-Gas Monitor were also obtained at several of the "cooks". Instantaneous levels of hydrogen chloride using that instrument were as high as 155 ppm during the extraction phase and frequently were above 50 ppm.

Iodine

Iodine samples were collected during four red phosphorous "cooks" and one hypophosphorous "cook". The levels measured in the cooking area ranged from 0.002 ppm to 0.15 ppm with levels at the remote area and in the breathing zone significantly lower (Table 7). Iodine is not used during the anhydrous ammonia method of methamphetamine production and therefore, samples for iodine were not taken during that method of production. Two samples exceeded the current ACGIH TLV level for iodine of 0.1 ppm as a ceiling limit.[12]

Airborne Methamphetamine

We detected methamphetamine residues on horizontal and vertical surfaces in most of the suspected methamphetamine laboratories, suggesting it was likely released as an aerosol during the production process. We hypothesized that the release occurred during the "salting-out" or precipitation phase. Our sampling efforts confirmed that methamphetamine is released during the extraction phase of methamphetamine production and is likely associated with the production of hydrogen chloride mist. The amount of methamphetamine released appears to be higher in the phosphorous based "cooks", although an interference was noted by the laboratory in the anhydrous ammonia "cooks" (Table 8). We believe that it is this release of methamphetamine that results in the majority of contamination that is found on surfaces within clandestine laboratories. In the "cook" area, the airborne levels of methamphetamine ranged from a low of 79 ug/m^3 to a high of 5500 ug/m^3. Even in some of the more distant sampling locations concentrations exceeding 4000 μg/m^3 were detected.

Table 7. Airborne iodine levels obtained during controlled cooks to determine potential exposures during clandestine methamphetamine manufacture (Scenario 2)

Manufacturing Methodology	Manufacturing Area (ppm)	Personal Sample (ppm)	Remote Area (ppm)
Red phos. #1	0.15	0.04	0.03
Red phos. #2	0.002	0.002	NA
Red phos. #3	0.12	NA	0.005
Red phos. #4	0.03	NA	0.005
Hypophos. #1	0.005	0.004	0.001

NA: not available; ppm: parts per million.

Methamphetamine Wipe Samples

A total of 53 surface wipe samples were taken for methamphetamine during this project (Table 9). All of the sam-

Table 8. Airborne methamphetamine levels

Manufacturing Method	Manufacturing Area (ug/m^3)	Remote Area (ug/m^3)
Red phos. #2	5500	4200
Red phos. #3	520	99
Red phos. #4	760	510
Anhydrous #1[a]	>680	>12
Anhydrous #2[a]	>79	>2.6
Anhydrous #3[a]	>170	>158
Hypophos. #1	3800	4000
Hypophos. #2	680	NA
Mean	1524 (2252)	1283 (2202)
Median	680 (760)	158 (2255)

NA: not available; ( ): calculation of mean and median results excluding the values for cooks using the anhydrous ammonia method of manufacture.
[a] Interference noted by analytical laboratory.

Table 9. Methamphetamine wipe sample results obtained during controlled cooks to determine potential exposures during the clandestine manufacture of methamphetamine (Scenario 2)

Distance from Cook (m)	Number of Samples	Mean (ug/100 cm^2)	Median (ug/100 cm^2)	Range (ug/100 cm^2)
Phosphorous cooks ($n = 5$)				
<2	14	100.9	21.5	0.1–860.0
2–4	11	40.7	19.0	0.8–45.0
>4	4	21.7	22.1	11.6–31.0
Anhydrous ammonia cooks ($n = 3$)				
<2	8	25.2	3.7	0.1–160
2–4	8	1.0	0.9	0.2–2.3
>4	8	0.4	0.2	0.1–1.2

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ples taken in the area of the "cook" (within that room or immediately adjacent rooms) were above the limit of detection. The concentrations were generally higher in "cooks" for the phosphorous methods of production and lower for the anhydrous ammonia method of production. As expected, concentrations of methamphetamine on surfaces close to the "cook" were higher than surfaces further from the immediate cooking area.

Various states in the US have developed re-occupancy criteria for surface contamination with methamphetamine, these criteria range from 0.1 μg/100 cm^2 to 0.5 μg/100 cm^2.[14] In our investigations, virtually all surfaces within a structure were found to be contaminated above 0.1 μg/100 cm^2 after a single "cook". Some areas close to where the "cook" occurred were contaminated with levels of methamphetamine that exceeded 100 μg/100 cm^2, a factor that is 1000 times current state based contamination criteria.

Contamination of Clothing

Methamphetamine was detected on the protective clothing of "cook" participants during many of the controlled "cooks" (Table 10). Surface wipes were collected on approximate 100 cm^2 areas on the clothing of the participants. Because wiping precise surface areas of protective clothing worn in the area was difficult, results should be interpreted in units of μg/sample rather than per 100 cm^2. The amount of methamphetamine present ranged from 0.2 μg/sample to a high of 150 μg/sample. Surface wipe concentrations on the back and head areas were more likely due to airborne methamphetamine than from splatter during manufacturing. The arithmetic mean level of contamination was 16.3 μg/sample and the median contamination was found to be 6.4 μg/sample. Virtually everyone participating in the extraction phase of the "cook" was contaminated. Most individuals, exiting the "cook" area prior to the extraction phase, did not have detectable contamination.

Table 10. Post cook clothing contamination for individuals participating in controlled methamphetamine cooks (Scenario 2)

Cook #	Cook Methodology	Location of Sample	Methamphetamine Level (ug/Sample)
1	RP	Front	16.0
1	RP	Head	16.0
1	RP	Front	18.0
2	RP	Front	8.1
2	RP	Back	4.9
2	RP	Front	14.5
2	RP	Back	2.5
2	RP	Front	10.3
2	RP	Back	6.0
2	RP	Front	9.0
3	AA	Front	150
4	AA	Front	58
5	AA	Front	0.3
6	RP	Front	6.4
6	RP	Back	4.6
6	RP	Front	9.4
6	RP	Back	1.6
7	RP	Front	3.7
7	RP	Back	1.1
7	RP	Front	1.8
7	RP	Back	0.2
Mean			16.3
Median			6.4

Front: sample taken on the front portion of the protective clothing worn by the participant; back: sample taken on the back portion of the protective clothing worn by the participant; RP: phosphorous methodology used in the controlled cook; AA: anhydrous ammonia methodology used in the controlled cook.

DISCUSSION

Based on our sampling results, the chemicals of greatest concern from a human health perspective include phosphine, iodine, ammonia, hydrogen chloride (all of which are very potent irritants), and of course methamphetamine itself. We show that concentrations for many of these chemicals approach or exceed current occupational exposure guidelines. This is especially true of exposures to phosphine, iodine, anhydrous ammonia, and hydrogen chloride. Each of these compounds may exceed the occupational exposure guidelines as set by the Occupational Safety and Health Administration and by the American Conference of Governmental Industrial Hygienists.[11,12]

Phosphine

During our controlled "cooks", phosphine was generated during the red phosphorous methamphetamine "cooks". Phosphine was produced at concentrations measured from 0.1 ppm to 13 ppm during the cooking phase. Phosphine was produced on all occasions during the "cook" and not just during an overheating event. The current ACGIH TLV for phosphine is 0.3 ppm on an 8-hour time weighted basis with a STEL of 1.0 ppm.[12] The highest level observed was 13 times the STEL, suggesting that overexposure to phosphine is likely.

Phosphine is a severe pulmonary irritant that may cause dyspnea, headache, paresthesia, diplopia, tremor, jaundice, and pulmonary edema. Death from exposure to phosphine used an insecticide has occurred in exposed persons.[15] Fatalities thought to be due to phosphine exposure were also linked to a methamphetamine laboratory in Los Angeles, CA where three persons were found dead in a motel room.[16] A laboratory investigator was also reported by Burgess[17] to have developed dizziness, dry cough, headache, and diarrhea, with a delayed onset of cough and dyspnea, after investigating a clandestine metham-

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phetamine laboratory. The exposure was measured at 2.7 ppm phosphine and the duration of exposure was approximately 20–30 minutes. These levels are in the same range as the levels measured during our investigation. In workers, phosphine exposure has been shown to cause gastrointestinal, respiratory, and central nervous symptoms at concentrations that are less than 10 ppm.[18]

There are a number of reasons why phosphine intoxication may be more common than reported. Phosphine does have a detectable odor but it may be less readily identified with the presence of the more odorous hydrocarbons present during the "cook". In addition, the pulmonary toxicity of phosphine may occur shortly after exposure or it may be delayed for 18 hours or more. These factors may result in fewer reported symptoms, although pulmonary irritation is a common complaint after a clandestine laboratory investigation.

Children and adults that are especially susceptible to pulmonary problems, such as asthmatics, individuals with chronic obstructive pulmonary disease, emphysema, etc, may show significantly greater effects to exposure levels of phosphine that are well below the concentrations allowed in the occupational environment.

Iodine

Airborne iodine concentrations ranged from 0.002 ppm to 0.15 ppm. The highest concentrations exceed the current ACGIH Ceiling TLV of 0.1 ppm.[12] The walls in many of the suspected "cook" areas had brownish-yellow stains that reacted with spray starch forming a dark blue color, an indicator for iodine.

Airborne iodine is a heavy halogen vapor considered to be more irritating and corrosive than bromine or chlorine gases. In animal studies, iodine vapor has been found to be intensely irritating to mucous membranes, causing damage in both the upper and lower portions of the respiratory tract. Iodine vapors can be an intense irritant to the eyes, mucous membranes and skin and have a steep effects curve in that concentrations of 0.1 ppm may cause very little effect while levels of 0.5 ppm may cause severe irritation.[19]

Although there have been no documented cases of over-exposure to iodine vapor in clandestine methamphetamine laboratories reported in the literature, iodine could be a plausible cause of mucous membrane and eye irritation reported at these investigations. Iodine may persist for some time in the walls, carpeting, draperies, and furnishings in many of these clandestine laboratories. The fact that it is commonly observed on the walls, even after months of no cooking, suggests that it can be very persistent.

Iodine persistence in the environment of the "cook", which is often the kitchen of the residence, results in an important potential exposure to the children that are present in the clandestine laboratories as well as children who inadvertently become residents in a building previously used as a methamphetamine laboratory. Children crawling on contaminated carpeting are likely to have exposures to surface concentrations of iodine.

Hydrogen Chloride

Hydrogen chloride was measured during all methamphetamine "cooks". Time-weighted average concentrations were in a range of 0.02 ppm to 20 ppm. The peak level measured during one controlled "cook" with the real-time monitor was 155 ppm. The current ACGIH TLV for hydrogen chloride is a ceiling value of 2.0 ppm, much lower than the levels found during the controlled "cooks" that we conducted.[12] The NIOSH Immediately Dangerous to Life and Health concentration for hydrogen chloride is 50 ppm, which was approached during the salting-out phase conducted during the controlled "cook".[13,20]

Exposures to hydrogen chloride can cause acute and chronic health effects. One individual exposed during a swimming pool cleaning effort developed severe bronchospasm and asthma. Workers exposed to as little as 10.7 ppm of hydrogen chloride experienced work impairment. Hydrogen chloride is a strong irritant of the eyes, mucous membranes, and skin at levels that are well below the levels that we have measured during our controlled "cooks". It would seem likely that individuals exposed to the measured concentrations that we have found would have acute symptoms from the exposure.[20]

Anhydrous Ammonia

The concentration of ammonia measured during anhydrous ammonia "cooks" was somewhat of a surprise since the levels, even as a time-weighted average during the "cook", approached or exceeded the Immediately Dangerous to Life and Health Levels published by NIOSH.[13] Anhydrous ammonia is an extremely irritating compound that poses an inhalation hazard, a dermal hazard, an ingestion hazard, and an ocular and mucous membrane hazard. The compound has a very pungent and suffocating odor that typically drives exposed individuals from the area. It is possible, however, that olfactory fatigue can set in quickly allowing increased exposures to individuals. At concentrations exceeding 50 ppm, individuals may experience nose, throat, eye, mucous membrane, and airway irritation. Extended exposure may cause wheezing, shortness of breath, and chest pain as well as tearing and ocular damage.[21]

Exposure to high levels of anhydrous ammonia (levels exceeding 2500 ppm) have been found to cause severe corneal irritation, difficulty breathing, bronchospasm, chest pain and pulmonary edema in otherwise healthy adults. The pulmonary edema associated with these exposures has been fatal in some instances. Repeated exposure to high levels of anhydrous ammonia may cause chronic cough, bronchitis, asthma, vocal cord dysfunction, reactive airways disease, and lung fibrosis. In some cases, a permanent decrement in pulmonary function has occurred due to anhydrous ammonia exposures. Contact with the liquid state may also cause serious eye injury or blindness as well as skin burns.[22]

The current OSHA Permissible Exposure Level is 50 ppm and the ACGIH TLV for ammonia is 25 ppm as an 8-hour time-weighted average and 35 ppm as a short-term exposure level (15 minutes or less no more than four times per day).[11,12] The AIHA Emergency Response Guidelines[23] suggest that most individuals can be

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exposed to 25 ppm of ammonia for at least 1 hour without suffering more than mild, transient health effects (ERPG-1). At 150 ppm, most individuals can be exposed for up to 1 hour without experiencing any irreversible or serious health effects (ERPG-2). At an exposure level of less than 750 ppm, most individuals could be exposed for up to 1 hour and not experience any life threatening health effects. The current NIOSH Immediately Dangerous to Life and Health Level is listed as 300 ppm.[13]

As indicated by the preceding information, anhydrous ammonia poses a significant potential health risk to exposed individuals. The levels of anhydrous ammonia observed during the "cook" ranged from 130 ppm to over 437 ppm as a time weighted average during the "cook". The real-time measurements of anhydrous ammonia were so high that we were unable to obtain a reliable quantification, however, the Drager tube readings indicated that levels of ammonia routinely approach 2000 ppm during the initial phases of the operation and may remain at over 500 ppm even in areas distant to the "cook". Based on these exposures, it is likely that individuals using this method of manufacturing methamphetamine will be over-exposed to anhydrous ammonia and that they will suffer some symptoms associated with that exposure.

Methamphetamine Exposures

Surface contamination throughout the buildings used to manufacture methamphetamine was a consistent finding. Even labs that had been shut down several months prior to testing had high contamination levels of methamphetamine present on many surfaces within the building. Samples as high as 16,000 μg/100 cm^2 were found in the actual laboratories, with most samples over 25 μg/100 cm^2.

Although the effects of methamphetamine are well known on individuals using the drug, the effects of low level exposures to emergency personnel or other associated individuals are not as well studied. It is known that methamphetamine may cause some teratogenic effects and may change behavior in exposed infants. Prenatal exposure to methamphetamine has been shown to cause an increase in pre-term labor, placental abruption, fetal distress, and postpartum hemorrhage.[24] Infants exposed to methamphetamine are generally smaller, have feeding difficulties, and are described as "very slow". Infants borne to mothers that have used methamphetamine during pregnancy may have abnormal sleep patterns, poor feeding, tremors, and hypertionia. In some reports, subtle neurological abnormalities have also been found.[24,25]

Surface contamination throughout the buildings used to manufacture methamphetamine was a consistent finding.

Our study has shown that a myriad of chemicals (and potential exposures) occur during clandestine manufacture of methamphetamine.

Currently, various state re-occupancy levels for a residence that has been used as a clandestine laboratory range from 0.1 ug/100 cm^2 to 0.5 μg/100 cm^2.[26] These concentrations have not been developed on human health exposure studies, rather, at the feasible limits of detection since no "safe" threshold level of exposures has yet been established. Methamphetamine aerosols can contaminate surfaces throughout a building when the drug is made and when it is used (smoked). It is difficult to determine dermal exposures and actual biological doses for individuals working or living within that atmosphere. It is therefore logical to assume that hand contamination can result not only in ingestion exposure, (especially in the case of children) but it may also contribute to systemic exposures by percutaenous absorption in both children and adults.

CONCLUSIONS

Our study has shown that a myriad of chemicals (and potential exposures) occur during clandestine manufacture of methamphetamine. Concentrations at these labs may approach IDLH levels, which by definition, is an extremely dangerous environment for investigating officers, the criminals themselves, and especially for children that might be present in the structure. Recent studies have shown that individuals responding to these clandestine methamphetamine laboratory investigations have an elevated risk of injury. Of 112 methamphetamine-associated hazardous materials events reported to the Centers for Disease Control, 53% resulted in injuries with 155 persons injured. The primary symptoms were respiratory irritation and eye irritation and, based upon the chemical exposures that we have found to be present within these laboratories, that is predictable.[1]

For law enforcement officers and other first responders, our results indicate that unless a suspected laboratory is first confirmed to be inactive, the minimum ensemble of personal protective equipment (PPE) should include complete barrier (skin) protection and the highest level of respiratory protection available, a self contained breathing apparatus (SCBA). In other words, all individuals entering a suspected laboratory should wear chemical-resistant clothing, gloves and boots, and an SCBA operating in the pressure demand mode.

If it is known that the laboratory is not in operation and has not been in operation in the recent past, then a lesser degree of respiratory protection may be used. We suggest a minimum of a properly fit tested, NIOSH approved full-face air purifying respirator. We suggest that the respirators be config-

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ured with chemical cartridges protective against acid gases, particulates, and hydrocarbons and that these canisters be discarded at the end of the day or after each laboratory investigation, whichever occurs first. Disposable, chemical-resistant clothing should be worn by all individuals since surface contamination from methamphetamine residues is almost guaranteed. Investigators should also be cautioned not to open sealed bags due to the potential of phosphine release from a "death bag" used to collect the phosphine.

Based on our studies, anyone entering a clandestine methamphetamine laboratory should assume that anything present within the laboratory is contaminated with methamphetamine and likely other chemicals. It is prudent to assume that anyone or anything removed from a lab requires decontamination to prevent subsequent contamination and possibly secondary exposures. Training should be provided to assure that officers are aware of the possibilities of contamination, the potential health effects, and the potential to carry exposures out of the laboratory and back to their own families.

The chemical contamination present within a building that has been used as a clandestine methamphetamine laboratory has special importance for children of a family associated with that structure. A report from the Colorado Department of Public Health and Environment suggests that an infant living in a structure that has been contaminated with the levels of methamphetamine that we have documented may experience serious health effects.[26] The report suggests that infants in these environments may have internal methamphetamine doses ranging from 0.41 mg/kg/day to 13.3 mg/kg/day which approach and may exceed the dose found in adults abusing the drug. Considering that some developmental and neurological endpoints to infants may exist at levels less than 0.01 mg/kg/day, these doses appear to be very important. In 2001, 2,028 children were documented to be present at seized clandestine methamphetamine laboratories and 700 of them tested positive to the drug itself.[27] Based on our findings, it is surprising that only 700 were positive. At this time, the authors are not aware of any prospective study that has looked at the potential health and behavioral risks to these children.

Study Limitations

This study was conducted under uncontrolled conditions in the field, frequently while wearing PPE under potentially dangerous conditions. Under these conditions, sampling can be difficult, equipment can malfunction, and obviously concentrations of chemicals are dynamic. The environmental sampling that we conducted at the suspected clandestine methamphetamine laboratories reveal very low concentrations of chemicals with the exception of the surface wipe samples. That should not be taken to imply that these conditions will always be representative. Chemical concentrations (and potential exposures) will depend upon the degree of laboratory activity, amount of building ventilation, manufacturing techniques and methodology used, equipment, and especially amounts and types of precursors utilized for each batch.

Results from the controlled "cooks" are expected to represent what might be considered "typical" for clandestine methamphetamine laboratory but, in fact, there may not be a "normal" or "typical" laboratory since many manufacturers may use significantly higher amounts of precursors in areas with very low ventilation rates. Readers should understand that chemical concentrations occurring in actual clandestine laboratories are not necessarily predictable. Because manufacturing conditions vary widely, chemical concentrations are also expected to vary accordingly.

Although our best methodology and laboratory analysis techniques were utilized during this study, some of the results may have been less accurate than we had hoped. The results of the phosphine sampling were plagued with high phosphine levels on the control samples suggesting that the analysis results were not accurate. In addition, real-time instruments, such as those used for phosphine and hydrogen chloride in the controlled "cook" may give results that are less accurate than those obtained using laboratory methods. These findings overall should serve to advise first responders on prudent safety precautions and should illuminate the potential hazard to unwitting victims, such as children who are present in these potentially dangerous environments.

ACKNOWLEDGMENTS

This project was sponsored, in part, by a grant from the Colorado Department of Public Safety and the United States Department of Justice through the Colorado Methamphetamine Program. In addition, the National Institute for Occupational Safety and Health provided a substantial amount of the laboratory analysis. The authors would also like to acknowledge the North Metro Task Force in Thornton, Colorado, and especially Cdr. Lori Moriarty, Sgt. Jim Gerhardt, and Lynn Riemer who allowed us to sample at a number of their clandestine laboratory investigations. Assistance was also provided by the Colorado Alliance for Drug Endangered Children and many of the emergency service agencies (police, fire, and EMS) in the Denver Metropolitan Area.

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