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Surface Contamination Generated by “One-Pot” Methamphetamine Production

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ABSTRACT: Methamphetamine production is the most common form of illicit drug manufacture in the United States. The “one-pot” method is the most prevalent methamphetamine synthesis method and is a modified Birch reduction, reducing pseudoephedrine with lithium and ammonia gas generated *in situ*. This research examined the amount of methamphetamine surface contamination generated by one-pot syntheses or “cooks”, as well as the effectiveness of hosing with water as a simplified decontamination technique, to assess associated public health and environmental consequences. Concentrations of methamphetamine contamination were examined prior to production, after production, and after decontamination with water. Contamination was qualitatively field screened using lateral flow immunoassays and quantitatively assessed using a fluorescence covalent microbead immunosorbent assay. Following screening, 0 of 23 pre-cook samples, 29 of 41 post-cook samples, and 5 of 27 post-decontamination samples were positive. Quantitatively, one pre-cook sample had a methamphetamine concentration of 1.36 ng/100 cm^2. Post-cook and post-decontamination samples had average methamphetamine concentrations of 26.50 ± 63.83 and 6.22 ± 12.17 ng/100 cm^2, respectively. While all one-pot methamphetamine laboratories generate different amounts of waste, depending on the amount of precursors used and whether the reaction vessel remained uncompromised, this study examined the surface contamination generated by a popular one-pot method known to law enforcement. By understanding the amount of surface contamination generated by common methods of one-pot methamphetamine production and the effectiveness of decontamination techniques used to remediate them, health risks associated with these production sites can be better understood and environmental contamination can be mitigated.

KEYWORDS: *one-pot, methamphetamine, surface contamination, decontamination, immunoassay*

1. INTRODUCTION

Methamphetamine (MA) is a schedule II stimulant that can be illicitly produced in clandestine laboratories by numerous methods. In the United States, the current most popular route of MA production is known as the “one-pot” method, which is a modified Birch reduction that uses sodium hydroxide and ammonium nitrate to produce ammonia gas *in situ* that can strip lithium of electrons and use them to reduce pseudoephedrine to MA (Figure 1);[1] these reactions take place within a single reaction vessel, giving it the name of the one-pot method.[2] The reactions that occur within the reaction vessel during one-pot MA production are shown in eq 1. These include: a) the reaction of ammonium nitrate with sodium hydroxide generates sodium nitrate, ammonia gas, and water; b) the reaction of lithium metal with ammonia gas, resulting in the liberation of an electron; c) the liberated election is used to reduce pseudoephedrine or ephedrine to methamphetamine, resulting in the formation of lithium hydroxide (or water if the hydroxyl leaving group reacts with a proton).

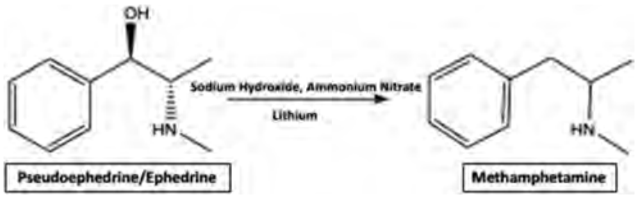

Figure 1. One-pot reaction used to reduce pseudoephedrine or ephedrine to MA.

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$$\mathbf{a.})\ NH_4OH + NaOH \rightleftharpoons NaNO_3 + NH_3 + H_2O$$

$$\mathbf{b.})\ Li \xrightarrow{NH_3} Li^+ + e^-$$

$$\mathbf{c.})\ C_{10}H_{15}NO + e^- + Li^+ + H^+ \rightarrow C_{10}H_{15}N + LiOH$$

With any method of illicit MA production, site contamination becomes an issue, which results in public health risks if the site is to be reinhabited. MA aerosols volatilized during production will settle on surfaces, leading to exposure risks for individuals entering and spending time within these areas. Additionally, organic solvent containing dissolved MA freebase may be spilled during production, or the bottle may rupture due to repeated overpressurization, causing localized areas to have relatively higher MA contamination.

Typical exposures associated with former MA laboratories include dermal, inhalational, and oral exposures.[3] Studies have shown that MA can penetrate human skin, with varying absorption rates that are dependent on the material of the contaminated surface, with smooth, nonporous surfaces resulting in greater rates of absorption when compared to rough, porous surfaces;[4] moisture and pH of the skin have also been found to impact the dermal absorption rate of MA.[5] Additionally, normal household activities, such as walking, can cause resuspension of MA particulates leading to risks for inhalation exposures.[6] The risks for oral exposures is greatest in children, who tend to have oral fixations and may put contaminated items, hands, and fingers in their mouths.[7]

While no work has examined surface MA contamination generated by one-pot MA laboratories, previous work by Martyny et al. has examined surface contamination from other methods of illicit MA production, namely, the red phosphorous (Red-P) and the Birch reduction methods. When examining the surface contamination generated by simulated MA productions, Martyny et al. reported concentrations ranging from 0.1 to 860 μg/100 cm^2 for Red-P production and from 0.1 to 160 μg/100 cm^2 for Birch reduction simulated production.[8] In seized MA laboratories, Martyny et al. found average surface MA concentrations of 499 μg/100 cm^2, exposure to which is equivalent to a dose of 0.38 mg/(kg day) MA when applied to Hammon and Griffin's model for dermal and oral MA exposure in infants;[8,9] CNS stimulation can occur in naïve users at doses as low as 0.07 mg/kg.[9] While concentrations of MA surface contamination found in Red-P and Birch reduction illicit manufacture can be on the order of milligrams per 100 cm^2, these types of laboratories, by design, can produce large amounts of MA when compared to one-pot manufacture, which are typically for personal use and generate less than 2 oz. of the drug.[2] This is driven, in part, by the increased amount of starting material used by these other methods, as Red-P cooks have been reported to start with 150 g of pseudoephedrine and Birch reduction cooks with 30 g of pseudoephedrine. In contrast, one-pot MA cooks commonly use as much pseudoephedrine as the manufacturer can obtain, which is limited to 3.6 g per day and 9 g per month by Federal law and is further restricted by some State laws.[10−12] Because of the lower amount of MA produced using the one-pot method, it is presumed that this method results in less surface contamination, although the surface contamination generated has not been reported in the literature.

This investigation reports on surface concentrations of MA detected following one-pot MA manufacture. After collecting surface swab samples, the site of production was decontaminated by hosing with water, and the concentration of surface contamination was reevaluated. As there are many variations of the one-pot, all utilizing different laboratory equipment, laboratory techniques, and amounts and types of starting materials, no one study can be representative of all one-pot MA laboratories. Due to this, this investigation chose a one-pot method that has been posted online, referenced to on several clandestine chemist chat sites, and seized in clandestine MA laboratories by law enforcement, as it was determined to be a popular method of MA synthesis. Understanding the concentrations of surface contamination generated using popular one-pot methods is important in understanding public health risks that are associated with entering and/or living in these clandestine drug laboratories.

2. MATERIALS AND METHODS

2.1. Reagents and Materials.

The following reagents used for MA production were purchased from commercial hardware stores and were not of laboratory grade in order to more closely mimic a clandestine MA laboratory that would be encountered in a real-world setting: ammonium nitrate from instant cold compress packs (GoGoods.com, Inc, Columbia, MD), Coleman camp fuel (Model, 5103B253; Coleman, Wichita, KS), Prestone premium starting fluid (Prestone Products Corp, Lake Forest, IL), sodium hydroxide from Drain Out Crystal clog remover (Summit Brands, Fort Wayne, IN), sulfuric acid from Rooto professional drain opener (The Rooto Corp, Howell, MI), and Great Value iodized salt and Equate mineral oil (Wal-Mart Stores, Inc, Bentonville, AR). A mixture of ground pseudoephedrine-HCL/ephedrine-HCL tablets were obtained from a government source. Lithium ribbon was purchased from Sigma (Sigma-Aldrich Corp, St. Louis, MO). The reaction vessels used were 32 oz. Gatorade bottles (PepsiCo, Inc., Purchase, NY).

Methamphetamine was obtained as a 1 mg/mL solution in methanol from Cerilliant Corporation (Round Rock, TX). Methamphetamine-BSA conjugates and monoclonal antibodies to methamphetamine were obtained from Arista Biologicals (Allentown, PA). All water used was >18 MΩ cm ultrapure (Milli-Q Integral, Millipore Corporation, Billerica, MA). MagPlex microspheres were obtained from Luminex Corporation (Austin, TX). Biotin-labeled anti-mouse IgG, 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (EDC), and *N*-hydroxysulfosuccinimide sodium salt (sulfo-NHS) were obtained from Pierce Biotechnology, Inc. (Rockford, IL). Streptavidin *R*-phycoerythrin (streptavidin *R*-PE) was obtained from Molecular Probes (Eugene, OR).

2.2. One-Pot Cooks.

In total, six one-pot MA "cooks" were conducted inside a 2.55 × 3.11 × 2.62 m (20.78 m^3) plastic garden shed to simulate an environment where MA may be illicitly produced. A plastic garden shed was chosen as a simulative environment, as the location where the one-pot MA cooks were being performed could not have a permanent structure built, and an isolated, condemned house could not be obtained. During the one-pot cooks, the front doors of the shed were left open as a safety precaution, and a firefighter was on standby in case of a flash fire. During the 3 days in which the one-pot MA cooks were performed, the temperature ranged from 10 to 20 °C, and winds fluctuated from 0.5 to 6.25 m/s. Of the six one-pot MA cooks performed, three of the cooks utilized starting fluid (ethyl ether) as an organic solvent, and three utilized camp fuel (light petroleum distillate) as an organic solvent. The cooks were performed over 3 days, each organic solvent being utilized in one cook per day; ether was used during cooks 1, 4, and 6 while camp fuel was used during cooks 2, 3, and 5. While there is no "typical" amount of precursor materials used in a one-pot MA laboratory, these cooks were performed by following a known one-pot recipe from online drug forums and using 600 milligrams (mg) of a crushed mixture of pseudoephedrine-HCl and ephedrine-HCl tablets as the starting material.

After precipitation of the MA salts, they were separated from the organic solvent and dried via vacuum filtration with a shop vacuum, as has been observed to occur in clandestine MA laboratories. The resulting salts were tested using the NIK Public Safety Narcotics Identification System presumptive colorimetric Test U: methamphetamine or MDMA (Ecstasy) (NIK Public Safety Inc, Jacksonville, FL) and a FirstDefender RMX RX2863 Raman spectrometer (Thermo Scientific, Waltham, MA). A dark purple colorimetric reaction and a positive Raman spectrum match to MA was confirmatory for successful conversion of pseudoephedrine/ephedrine to MA. Following production, all reagents, labware, etc. were disposed of in a manner deemed suitable by the Oklahoma Bureau of Narcotics and Dangerous Drugs.

2.3. Surface Sampling and Analysis. Surface MA contamination was analyzed using two methods: MA specific lateral flow immunoassays (LFIAs) for near-real-time screening in the field and a fluorescent covalent microbead immunosorbent assay (FCMIA) for laboratory quantitation. A detailed outline of the sample collection procedures can be found in Section S2.3 of the Supporting Information.[6,8,13,14] The competitive lateral flow immunoassays (LFIAs) used in this research were developed and provided by the Health Effects Laboratory Division at the Centers for Disease Control and Prevention's National Institute for Occupational Safety and Health (CDC-NIOSH, Cincinnati, OH) and are available commercially through SKC (SKC, Inc, Eighty Four, PA); this sampling methodology has been previously described and reported, and the details can be found in Section S2.3.1 of the Supporting Information.[14] A slightly modified version of a fluorescence covalent microbead immunosorbent assay (FCMIA) developed by Smith et al. in 2010 was used to quantitate the amount of MA collected by each surface swab, and the details can be found in Section S2.3.2 in the Supporting Information.[15,16]

3. RESULTS AND DISCUSSION

3.1. Competitive Lateral Flow Immunoassays. In total, 96 qualitative LFIAs analyses were performed: 23 pre-cook, 41 post-cook, 27 post-decontamination (post-decon), and 5 "other" samples; the five other samples included 2 field blanks and 3 samples used to ensure the absence of MA contamination from the standard clothing worn by those collecting samples throughout the investigation. Pre-cook samples were only collected on the first day, prior to any MA production; post-cook samples were collected following each MA cook, and post-decon samples were collected after the researchers and/or cook shed were hosed with water. Table 1 summarizes the results of the LFIA analyses; each individual LFIA analysis' results are provided in the supplementary data (Tables S1−S5). All pre-cook and other LFIAs were negative. For the post-cook samples, 24 were positive, and 5 were trace positives (only a faint line was observed at the "T" location on the cassette; the line was not distinct enough to be deemed a true negative); 12 samples were negative. For the post-decon samples, 2 were positive, and 3 were trace positives; 22 were negative.

Table 1. Summary of the LFIA Results[a]

	positive	trace	negative	total
pre-cook	0	0	23	23
post-cook	24	5	12	41
post-decon	2	3	22	27
other	0	0	5	5
total	26	8	62	96

[a] Pre-cook samples were collected prior to any MA production. Post-cook samples were collected after MA production, and post-decon samples were collected after hosing the researchers and the cook shed with water. Trace positives had a faint line present at the test site of the LFIA, but the line was not distinct enough to be deemed a true negative.

3.2. Fluorescent Covalent Microbead Immunosorbent Assay. A summary of the FCMIA quantitative data is shown in Table 2, and individual quantitative results are shown in Tables S1−S5. The instrumental MA background in pre-cook samples acting as sample blanks was observed at an average concentration of 0.33 ng/100 cm^2, so an administrative limit of detection (LOD) was set at 1 ng/100 cm^2 (3× the background concentration); all results below 1 ng/100 cm^2 were designated as "not detected" (ND). Only one pre-cook sample had MA present at a concentration greater than 1 ng/100 cm^2. This sample was collected from the top of the table where the one-pot cooks took place, in the sample location closest to the back to the shed (sample site 4 in Figure S1). The sample was analyzed and determined to have 1.36 ng/100 cm^2 of MA, and the two swab samples collected adjacent to this sample (and shipped together to the Taft Laboratory) were analyzed and reported at 257.69 and 130.82 ng/100 cm^2, respectively; it is reasonable to assume that cross-contamination occurred during shipping or, more likely, during analysis. Cross-contamination during shipping or analysis of samples would also explain the background MA concentration observed.

Table 2. Summary of the FCMIA Quantitative Data (All Concentrations Are in ng/100 cm^2)

	pre-cook	post-cook	post-decon	other
max concn	1.36	264.35	45.67	1.62
min concn	N/A	1.18	1.04	N/A
mean concn	N/A	26.50 (±63.83)	6.22 (±12.17)	N/A
median concn	N/A	5.40	1.67	N/A
% ND	95.7%	17.1%	22.2%	80.0%

Post-cook samples had a maximum MA concentration of 264.35 ng/100 cm^2 and a minimum MA concentration of 1.18 ng/100 cm^2, with a mean concentration of 26.50 ± 63.83 ng/100 cm^2. Of the 41 samples collected post-cook, 7 (17.1%) had undetectable concentrations of MA. The swab samples containing the highest concentration of MA were collected from the top of the table where the one-pot MA cooks were being performed and from the PPE of the researchers performing the cooks. While these results are likely explained by these samples being collected close to where the reaction vessel was located throughout the cook, it is more likely that the higher MA concentrations were the result of spilled organic solvent, from the filtration process, which contained MA freebase dissolved within it. If the relatively higher surface concentrations were from being in close proximity to the one-pot, it could be expected that the sleeves of the researchers' PPE would have higher MA concentrations, but the PPE with the highest MA concentration was the legs and belt, which is more suggestive of a solvent splash or leaning against a contaminated table. Additionally, two of the camp fuel cooks resulted in bottle failures due to overpressurization of the reaction vessel. This caused organic solvent to be spewed across the table and on the researcher's lower body, further suggesting that a majority of the MA surface contamination generated by one-pot MA laboratories results from spillage of organic solvents during or post-cook, though the increased MA concentration throughout the entirety of the shed also suggests that some contamination is due to MA aerosolization. While it is unknown how common bottle failures are within one-pot MA laboratories, 15% of laboratories discovered by law enforcement are due to fires resulting from bottle failures, suggesting that bottle failures are not an uncommon occurrence.[17]

Post-decon samples had a maximum MA concentration of 45.67 ng/100 cm^2 and a minimum MA concentration of 1.04 ng/100 cm^2, with a mean concentration of 6.22 ± 12.17 ng/100 cm^2. Of the 27 post-decon samples, only 6 (22.2%) had undetectable concentrations of MA, but all post-decon samples had lower concentrations of MA when compared to the same locations tested post-cook. While most of the sample sites still had quantifiable amounts of MA present after the wet decontamination process, all but three had MA concentrations below 5 ng/100 cm^2, and all were below 50 ng/100 cm^2, which is the lowest remediation concentration required in the United States.[18] The three higher MA concentrations were all located on the cook table; two were collected following the two bottle failures, and the third was collected on the same day as the second bottle failure, following the second cook of the day. It is believed that when the organic solvent spilled during the bottle failures, it deposited a significant amount of MA on the sample sites, and that the hydrophobicity of the solvent prevented the MA from being washed away during the water hosing decontamination process, instead leaving a thin film on the sample sites that contained dissolved MA. The use of soap, as suggested by EPA for remediation of MA laboratories, may have aided in decontaminating these sites by breaking down the organic solvent and allowing the water to wash the deposited MA off the contaminated sites.[19]

Figure 2 shows a box and whisker plot comparison of the observed post-cook and post-decon MA concentrations. The box portion of each plot depicts the first quartile, median, and third quartile MA concentrations. The whiskers depict the values that fall within 1.5 interquartile ranges (IQRs) above the third quartile and below the first quartile. The "×" on each plot depicts the mean MA concentration from each sampling time-point. As can be seen, all post-decon MA concentration 1.5 IQRs above the third quartile (4.30 ng/100 cm^2) are lower than the median post-cook MA concentration (5.40 ng/100 cm^2), and the post-decon median MA concentration (1.67 ng/100 cm^2) was lower than the first quartile post-cook MA concentration (2.50 ng/100 cm^2). This demonstrates that a simple water hosing was effective at decontaminating most of the cook site and PPE (median reduction of 70%) during this research, but not all of it. The three post-decon samples with MA concentrations greater than 1.5 IQRs above the third quartile suggest that a simple water hosing is not as effective in decontaminating locations where organic solvent was spilled, and a more robust decontamination method is needed in these locations. This data shows the importance of post-decon testing in multiple areas within sites of MA production, with focal points being locations where it is suspected that a one-pot was actually performed, as these locations will likely have a higher MA surface contamination prior to decontamination and may require more robust decontamination techniques to remediate to an acceptable concentration.

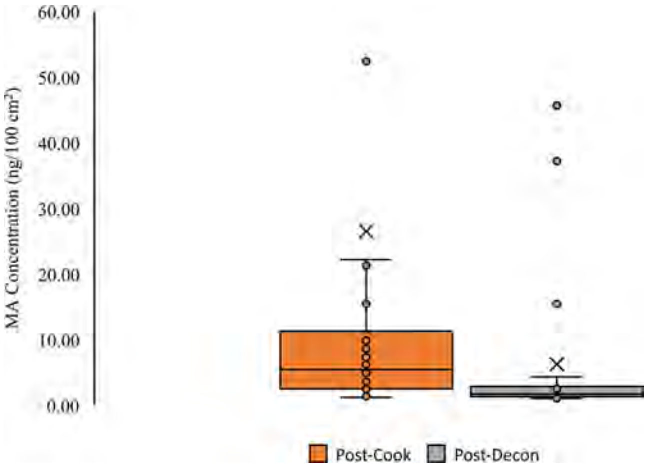

Figure 2. Distribution of observed MA concentrations post-cook and post-decon. The ends of the boxes define the 25th and 75th percentiles while the line in the box defines the median, which was not included in the quartile calculations. The "×" depicts the mean MA concentration observed from each sampling time-point. The error bars represent 1.5 IQRs above the 3rd quartile or below the 1st quartile. Three points from the post-cook samples are not shown to allow for better visualization of the plots and include MA concentrations of 130.82, 257.69, and 264.35 ng/100 cm^2.

As previously mentioned, Martyny et al. examined the amount of MA surface contamination observed following two other routes of MA production: Red-P cooks and Birch reduction cooks.[8] When compared to these two methods of MA production, the one-pot produced a much lower concentration of surface contamination. At a distance of less than 2 m from the cook, which was the distance at which all of the one-pot samples were collected during this study, Red-P cooks had a mean concentration of 100 900 ng/100 cm^2; Birch reduction cooks had a mean concentration of 25 200 ng/100 cm^2, and the one-pot cooks had a mean concentration of 26.50 ng/100 cm^2.[8] One reason for this difference in MA surface contamination concentrations stems from the amount of pseudoephedrine the cooks began with, as Red-P cooks have been reported to start with 150 g of pseudoephedrine and Birch reduction cooks with 30 g, and the one-pots performed in this study only used 0.6 g of pseudoephedrine.[10,11] By starting with less pseudoephedrine than other methods, the one-pot produces less MA and thus cannot produce as much surface contamination. Because of the relatively small size of one-pot MA cooks, clandestine laboratories that use this method generally contain multiple reaction vessels, with reports of laboratories containing as many as 100 one-pots.[20] Due to this, surface sampling from actual locations of clandestine one-pot MA manufacturing may provide surface contamination results that closer resemble those found by Martyny et al.

3.3. LFIA and FCMIA Comparison. When comparing the qualitative results of the LFIA to the quantitative results of the

Table 3. Number of True Positives, False Positives, False Negatives, and True Negatives Observed with LFIA When Compared to FCMIA When the LOD Was Set at 1 ng/100 cm^2 (Left) and 2 ng/100 cm^2 (Right)

LOD=1 ng/100 cm^2		FCMIA	
		Positive	Negative
LFIA	Positive	33	1
	Negative	23	39

Sensitivity	58.93
Specificity	97.50

LOD=2 ng/100 cm^2		FCMIA	
		Positive	Negative
LFIA	Positive	31	3
	Negative	4	58

Sensitivity	88.57
Specificity	95.08

FCMIA, 1 false positive and 23 false negative results were observed (Table 3, left). The high frequency of false negatives stems from the difference in detection limits; the LFIA was rated for 50 ng/100 cm^2 while the FCMIA had an LOD of 1 ng/100 cm^2. While the LFIA was rated to have an LOD of 50 ng/100 cm^2, this rating is based on the ability of individuals inexperienced at swabbing surfaces for drugs, and on the ability of the assay to detect MA at 50 ng/100 cm^2 from multiple surface materials and textures.[14,21] This means that the actual LOD of the LFIA is likely much lower, and this investigation found the apparent LOD around 2 ng/100 cm^2. If the FCMIA LOD was adjusted to 2 ng/100 cm^2, the LFIA resulted in just 3 false positives and 4 false negatives out of 96 samples, with a sensitivity of 88.57% and a specificity of 95.08% (Table 3, right).

4. CONCLUSION

This study sought to determine the concentration of MA surface contamination observed following one-pot production. Following production, the presence of MA surface contamination was observed in near-real time with the use of LFIAs. While claiming to have a limit of detection of 50 ng/100 cm^2, it was found that the LFIAs were able to consistently detect MA at concentrations as low as 2 ng/100 cm^2. Using FCMIA, MA surface contamination concentrations within the cook site were found to range from <1.00 to 264.35 ng/100 cm^2 following one-pot production. While the higher MA concentrations observed exceed the remediation standards in many states, it is well below the concentration of surface contamination measured following Red-P and Birch reduction MA cooks.[8,18] The lower concentration of MA surface contamination observed following the one-pot cooks was attributed to the lower amount of pseudoephedrine used during the one-pot MA production. Additionally, this study measured the concentration of MA surface contamination after single one-pot cooks while illicit one-pot MA laboratories generally contain multiple reaction vessels. This suggests that the surface contamination concentrations in actual clandestine one-pot MA laboratories may be much greater than the concentrations observed during this research.

When using just water as a simple decontamination agent, there was a significant amount of MA removed from the cook site, except for areas where organic solvent had been spilled (i.e., where bottle failures occurred during the cook). This highlights the importance of sampling multiple locations within a former MA laboratory before determining if it is safe for inhabitation. Focal points for surface sampling within former one-pot MA laboratories should be locations where the one-pot cook took place, as these locations are likely to have higher MA concentrations and may have been subjected to more organic solvent spillage than the rest of the laboratory. Due to this, these locations may need more robust decontamination techniques to reach acceptable remediation values. One limitation of this study was that it only attempted to decontaminate the nonporous high-density polyethylene (HDPE) of the shed and table, as well as the PPE worn by the research group. Further studies would be needed to test the effectiveness of water in decontaminating more absorbent materials, such as carpet, dry wall, and wood. Additionally, this investigation is not representative of the contamination level within a clandestine one-pot MA laboratory but rather examined the contamination generated by a single one-pot MA laboratory. To better represent actual clandestine one-pot MA production, surface sampling should be performed within these sites after they are seized by law enforcement. Alternatively, this study could be repeated with more one-pot MA laboratories being performed without the decontamination step between each cook, allowing for surface contamination to build up as additional cooks are complete.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.chas.0c00078.

Sample collection methodology, LFIA and FCMIA methods, and all test sites and their associated LFIA and FCMIA results (PDF)

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Notes

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