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Document Title:	Detection of One Pot Methamphetamine Laboratories via Wastewater Sampling
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Document Number:	254674
Date Received:	April 2020
Award Number:	IAA 2016-DNR-5470 (DOE SRNL)

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Detection of One Pot Methamphetamine Laboratories via Wastewater Sampling

Authors: Austin Ciesielski, Allison Veitenheimer¹, Jarrad Wagner¹, and David Pretorius²

Executive Summary

A total of 105 wastewater samples were collected in Oklahoma (OK), South Carolina (SC), and Georgia (GA) and analyzed by liquid chromatography tandem mass spectrometry (LC-MS/MS) at the Oklahoma State University Forensic Toxicology and Trace Lab (OSU-FTTL) for the presence of methamphetamine (meth), pseudoephedrine, amphetamine and the One Pot byproduct, 1-(1,4-cyclohexadienyl)-2-methylaminopropane (CMP). Grab and 24-hr composite samples were collected at gravity fed manholes, lift stations, and two wastewater treatment plants (WWTPs) in multiple communities. Multiple samples presented well above the upper limit of linearity of 300 ng/L and appear to range into the nanogram per milliliter level. Observed nanograms per milliliter meth accompanied by nanograms per milliliter amphetamine seen among samples suggest a biological origin. The highest meth values reported occurred over a weekend in samples taken at a gravity manhole fed by approximately 30 residencies; these high meth values were accompanied by noticeably low pseudoephedrine values and a lack of CMP, suggesting that during the time of sample collection, meth was being used, but not produced by One Pot chemistry. A grab sample meth value ranging into the ng/mL level from a mid-block manhole presents evidence of meth use narrowed to within approximately 15 residencies in the upper half of this block. This finding is also consistent with samples from consecutive manholes, suggesting meth consumption may be geographically narrowed in some cases to the connected residencies between manholes (where accessible). Consistent evidence was found for multiple-day persistence of target compounds (up to 12 days) at ambient temperatures while remaining in undisturbed open collection bottles. Wastewater testing for CMP at very trace levels was found to be a possible indicator of fixed localities with One Pot meth consumption by users or production by cooks, as 5 samples in the study were positive for CMP at a concentration below the CMP lower limit of quantitation of 50 ng/L.

Composite sample collection of stormwater in one residential area suggests a background level of contamination in the low hundreds of nanograms per liter may exist for the sampled neighborhood drain field, presumed as surface water runoff from roofs, cars, driveways, streets, etc.

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A handheld Photoionization Detector (PID) placed in a lift station headspace readily detected volatiles from a small deposit of camp fuel, representative of One Pot liquid waste sink-deposited to gravity drain approximately 50 feet upstream from the lift station.

Multiple findings of meth in neighborhood sewage water at apparent nanogram per milliliter levels is significant in opening the possibility for detection via portable, commercial off-the-shelf instruments with development of simplified field sample preparation steps.

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Introduction

The illicit production and use of methamphetamine is a problem that the United States has been combating for over 50 years.¹ As new legislation has been adopted to prevent methamphetamine production and hinder its availability for use, methamphetamine producers have developed new methods of production to get around such legislation. According to the United States Drug Enforcement Agency (DEA), the current favorite method of methamphetamine production in clandestine laboratories is the One Pot method, which accounted for 86% of all clandestine methamphetamine laboratory seizures in 2016.²

The One Pot method is a variation of older lithium-ammonia reduction methods that simplifies methamphetamine production to a single reaction vessel, which is commonly a plastic bottle. Lithium-ammonia reduction methods of methamphetamine production, such as the One Pot method, use lithium as an electron source to reduce the hydroxyl group on pseudoephedrine or ephedrine, forming methamphetamine. Ammonia acts as a solvent for the electrons, carrying them to the pseudoephedrine or ephedrine molecules.³ While older lithium-ammonia reduction methods used liquid ammonia to carry the electrons, in the One Pot method, ammonia gas is generated within the reaction vessel by combining sodium hydroxide and ammonium nitrate.

One reason the One Pot method has become the favorite method for methamphetamine production is its simplicity. To make a One Pot meth lab, the chemicals previously described can simply be added to a plastic bottle with camp fuel or ether and then allowed to react or "cook"; little chemistry knowledge is required to produce consumable methamphetamine. Additionally, the materials used to perform this process can be easily purchased at convenient stores without raising the suspicion of law enforcement that illicit methamphetamine production is occurring. Upon completion of the One Pot methamphetamine cook, the producer is left with solid waste, liquid waste, and the desired methamphetamine powder. The solid and liquid waste may be disposed of by throwing it in municipal trash, burning it, or dumping it down a drain.

In the United States, 76% of residencies are connected to public wastewater systems.⁴ If waste from a clandestine One Pot methamphetamine lab is dumped down a drain in a residence, it will enter the wastewater collection system and may then be collected and analyzed for chemical markers unique to One Pot methamphetamine production without the necessity of obtaining a search warrant.⁵ Such analyses were proven successful by the Oklahoma State University Forensic Toxicology and Trace Laboratory (OSU-FTTL) during a proof-of-concept study funded by the National Institute of Justice (NIJ) during FY16.⁶ This study is a continuation of the former proof-of-concept study, and aimed to apply the methods developed in the 2016 study to field applications. Wastewater samples were collected from municipalities in South Carolina, Georgia and Oklahoma, and were analyzed via solid phase extraction (SPE) followed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) for the presence of waste products from One Pot methamphetamine labs. These waste products include: methamphetamine, pseudoephedrine/ephedrine, and an over-reduced methamphetamine

byproduct known as 1-(1,4-cyclohexadienyl)-2-methylaminopropane (CMP). The methamphetamine metabolite amphetamine was also monitored to aid in differentiating methamphetamine production from methamphetamine use.

Materials and Methods

Sample Collection

Wastewater samples were collected in South Carolina, Georgia and Oklahoma. All samples collected from SC-GA were overnight shipped on ice to the OSU-FTTL in Tulsa, OK for analysis. Samples collected in Oklahoma were immediately taken to the OSU-FTTL for analysis after collection. Samples were collected from an array of wastewater lift stations, sanitary sewer lines accessed via manhole covers, and wastewater treatment plants. Sampling locations were a mixture of random "snapshot" locations, used to find evidence of methamphetamine use or production in select neighborhoods, as well as strategically planned sampling, including areas that had former One Pot methamphetamine laboratories. Collection procedures differed slightly between SC-GA and Oklahoma and are summarized below.

SC-GA Sample Collection Procedures

Samples SC 1-73 were collected using either an ISCO 3700 or ISCO 6700 automated portable sampler (Teledyne ISCO, Lincoln, NE) drawing wastewater by peristaltic pump through sanitary-grade Tygon[®] 3350 silicone tubing (Saint-Gobain, Malvern, PA) into 1000 mL polypropylene bottles (See Figure 1). Automated daily composite samples were comprised of approximately 15 mL hourly samples collected over 24 hours, remaining at ambient temperature until retrieval. Prior to analysis, grab and composite samples were transferred to 250 mL Nalgene high-density polyethylene sample bottles (Nalge Nunc International, Penfield, NY), cooled on ice, and shipped overnight to OSU-FTTL for laboratory analysis.



Figure 1. (Left) ISCO 3700 peristaltic pump autosampler typical of those used to collect grab and composite samples for SC 1-73. (Right) Collected samples remained at ambient temperature within the sampler housing for up to 12 days prior to being placed on ice in small coolers for shipment to OSU-FTTL for analysis.

SC 1-18 are neighborhood lift station grab samples collected from over one-third of the lift stations across a city of 30,000 residents. Neighborhoods represented by these samples range from dozens to hundreds of occupancies, with sample SC 2 being a mixed residential-industrial lift station that primarily serves an industrial park. Gravity-fed lift stations are denoted as "neighborhood" lift stations in Table 7, "multiple neighborhood" lift stations denote those receiving upstream flow from forced mains. For example, SC 13 lift station includes feeder flows from SC 8, 10, 12, 14, 15, 16, 17, 18, plus additional neighborhood lift stations not sampled.

SC 19-28 are grab samples collected the same day: SC 19, 20 and 21 are from 3 lift stations, each handling roughly one-third of the flows from a city of 30,000 and SC 22 is the confluence of these flows as they arrive at a wastewater treatment plant (WWTP). SC 23 is the arrival at the same WWTP of all flows from a second city of approximately 20,000 residents. SC 24 grab sample was taken from the combined WWTP inlet flow stream from all sources, totaling approximately 13 million gallons per day, which includes both cities, plus a few additional connected wastewater customers. This wastewater is mechanically aerated for microbial digestion in a large open basin for approximately one to two days, flowing continuously over a weir (SC 25) into a second large aeration basin (SC 26) of equal size for an additional 2-3 days residence time before flow to secondary clarifiers (SC 27) and a post-final treatment, composed of chlorine shock and removal. SC 28 was collected after final treatment and immediately prior to an outfall discharge piped to an adjacent river. Samples SC 22-28 are shown in Figure 2.



Figure 2. SC 22-28 grab samples collected from a county WWTP. The left three samples were collected prior to wastewater treatment, the next three were collected during different steps of the treatment process, and the right-most sample was taken post-treatment.

SC 29-35 are 24-hr composite samples taken from a lift station gravity-fed from a county detention center.

SC 36-46 are 24-hr composite samples taken from a municipality of approximately 5,000 residents. SC 36-40 are from two residential lift stations, with SC 36-38 from a neighborhood reported to have been the site of a former One Pot methamphetamine lab within the calendar year. SC 39 was taken from contained spillage observed within the wastewater autosampler housing arising from overfill of SC 36-38 collection bottles (See Figure 3). SC 41-46 are composite samples from the small municipality WWTP inlet, which includes flows from other lift stations in addition to the two sampled. A VOC-TRAQ II (MOCON, Inc, Lyons, CO) battery-powered diffusion photoionization detector (PID) equipped with a 10.6 eV lamp (See Figure 4) was also deployed in the lift station headspace over a period of six days during collection of SC 36-39 samples, to detect changes from background for volatile organic compounds (VOCs) present within the sewer.



Figure 3. Excess spillage from SC 36-38 was contained within the autosampler housing, collected, and reported as SC 39.



Figure 4. Diffusion photoionization detector powered via USB cable from a power tool battery. This assembly was suspended in the lift station headspace during collection of wastewater samples SC 36-39.

SC 47-51 are 24-hr composites from a lift station collecting from many thousands-ofresidencies. SC 51 sample was an attempt to establish a sample location on a hundreds-ofresidencies gravity-fed neighborhood tie-in just prior to its co-mingling with the lift station high inlet flow. This attempt proved unsuccessful, as the sampler inlet tubing was observed swept into the higher flow upon return a week later and the composite sample's precise origin is unknown.

SC 52-56 are 24-hr composite samples from a storm sewer access mistaken for a sanitary sewer manhole at a city park; the storm sewer manhole cover was within ten meters of the sanitary sewer manhole cover. As can be seen in Figure 5, collected storm sewer water presented observable clarity in comparison to sewage water samples.



Figure 5. Stormwater composite samples collected from a city park show greater visual clarity than sewage water collections.

As shown in Figure 6, SC 57-69 are 24-hr composite samples from a manhole at the end of a one-city-block headwaters gravity line connected to approximately 30 residencies in total. SC 73 is a grab sample from a small manhole, shown in Figure 7, accessed at the midpoint of this city block, noticeably too shallow and narrow to house the wastewater sampler for composite collection.



Figure 6. Composite samples SC 57-69 were taken over multiple days at the gravity fed manhole at the end of one city block of approximately 30 residencies with no other known feeders. The ISCO 3700 autosampler was positioned inside the manhole throughout sample collection.



Figure 7. Shallow manhole used to obtain grab sample SC 73 located one-half block upstream from the SC 57-69 samples.

SC 70-72 were collected from a manhole containing joining gravity flows from several dozen residencies. SC 70 was taken from a 5-day immersion of a polypropylene ALLWIK Absorbent Sock (Brady SPC, Milwaukee, WI) alongside SC 71 from 2 sorbent pads (New Pig Corporation, Tipton, PA). These sorbents are shown in Figure 8.



Figure 8. Polypropylene industrial sorbent sock (SC 70) deployed alongside sorbent pads (SC 71) in gravity manhole over multiple days. The left image shows the sorbents prior to being submerged into the wastewater flow and the right image shows the sorbents following retrieval.

OK Sample Collection Procedures

Water samples were collected by lowering a clean container into the wastewater until the container became full. Once full, the container was brought back to the surface and approximately one liter of the sample was poured into 1 L Nalgene wide-mouth high-density polyethylene bottles (Thermo Fisher Scientific, Waltham, MA). The collection container was rinsed three times with water prior to reuse for another sample. Samples were placed on ice while additional samples were collected. Once all samples had been collected, they were brought to the OSU-FTTL and immediately underwent cleanup procedures.

Wastewater Sample Cleanup

Upon receipt at the OSU-FTTL, samples were kept on ice until undergoing a cleanup procedure. If samples were not going to be cleaned the day of arrival in the lab, they were frozen at -20°C until the day prior to cleanup, at which time they were permitted to slowly thaw in a 4°C refrigerator.

To remove debris from the wastewater samples, 35 mL of sample was added to two 50 mL conical-bottom centrifuge tubes (VWR, Sugar Land, TX) (70 mL total). The tubes were then centrifuged at 2800 RCF for 8 minutes. After centrifugation, the samples were poured through a coffee filter (Farmer Bros. Co, Ft. Worth, TX) into a graduated cylinder until 50 mL of sample had been collected. The cleaned samples were then transferred to 250 mL TraceClean wide mouth amber glass jars (VWR, Sugar Land, TX) and stored in a 4°C refrigerator overnight.

Liquid Chromatography-Tandem Mass Spectrometry Method

Shimadzu UFLC pumps paired with an Applied Biosystems 4000 Q Trap MS/MS was used for the LC-MS/MS analysis (See Figure 9).



Figure 9. LC-MS/MS instrumentation setup at OSU-FTTL. Shimadzu UFLC pumps paired with an Applied Biosystems 4000 Q Trap MS/MS.

For liquid chromatography, chromatographic separation was achieved with a Chromegabond WR C18 5 µm column (15 cm x 2.1 mm) (ES Industries, Inc, West Berlin, NJ) with a Restek Raptor Biphenyl 2.7µm guard cartridge (5 x 3.0 mm) (Restek Corporation, Bellefonte, PA). Mobile Phase A consisted of 2 mM ammonium formate and 0.1% formic acid in LC-MS grade water, while Mobile Phase B consisted of 2 mM ammonium formate and 0.1% formic acid in LC-MS grade methanol. Ammonium formate was purchased from Alfa Aesar (Alfa Aesar, Ward Hill, MA). Formic Acid was purchased from EDM (EDM Millipore Corp, Billerica, MA). Methanol was purchased from JT Baker (Avantor Performance Materials Inc, Center Valley, PA). Nanopure water was obtained using a Barnstead Nanopure Diamond laboratory water system (Thermo Scientific, Waltham, MA). The LC had a total flow rate of 0.400 mL/min. Mobile Phase B concentration was held at 27.5% for the first 5 minutes of the sample run, and was then increased to 100% for 1.5 minutes, and was then decreased to 27.5% for 1.5 minutes for a total run time of 8 minutes. All changes in mobile phase B concentration were set to immediately

occur and end with no ramp. Injections were set at 1 μL and the oven temperature was set to 30°C.

For mass spectrometry, Table 1 shows the ion transitions and mass spectrometer voltage parameters for the compounds of interest. Amphetamine, amphetamine-d₆, methamphetamine, methamphetamine-d₅, and 1S,2S(+)-pseudoephedrine standards were all purchased at a concentration of 1 mg/mL from Cerilliant (Cerilliant Corp, Round Rock, TX). Pseudoephedrine-d₃ HCl standard was also bought from Cerilliant at a concentration of 100 µg/mL. CMP-HCl standard was purchased at a concentration of 1 mg/mL from Cayman Chemical (Cayman Chemical, Ann Arbor, MI). For simplicity, the naming of all analytes will be as follows: methamphetamine, pseudoephedrine, amphetamine, and CMP. Additionally, there are three deuterated internal standards, methamphetamine-d₅, pseudoephedrine-d₃, and amphetamine-d₆. Since this method cannot differentiate the diastereomers pseudoephedrine and ephedrine, all values reported for pseudoephedrine may also be contributed to by the presence of ephedrine. Pseudoephedrine is the only compound named for simplicity.

Table 1. Mass Spectrometry Parameters. Target analytes Methamphetamine, Pseudoephedrine, Amphetamine, and CMP were identified using two mass ion fragments each. Internal standards Methamphetamine-d5, Pseudoephedrine-d3, and Amphetamine-d₆ were identified using one mass ion fragment each. The values listed in column "Q1 Mass" are the molecular masses of each compound measured in Daltons. The values listed in column "Q3 Mass" are unique fragment ion masses measured in Daltons. The columns labeled "DP", "CE", and "CXP" refer to the voltages utilized for declustering potential, collision energy, and collision energy speed, respectively.

Compound	Q1 Mass (Da)	Q3 Mass (Da)	DP (volts)	CE (volts)	CXP (volts)	RT (min)
Methamphetamine	150.100	91.000	56.000	25.000	14.000	3.03
	150.100	119.000	56.000	15.000	4.000	
Methamphetamine-D5	155.000	91.100	60.000	20.000	4.000	3.03
Pseudoephedrine	166.132	91.099	46.000	39.000	12.000	2.24
	166.132	132.884	46.000	31.000	20.000	
Pseudoephedrine-D3	169.200	151.040	26.000	21.000	26.000	2.24
Amphetamine	136.200	119.000	36.000	13.000	18.000	2.89
	136.200	91.000	36.000	25.000	14.000	
Amphetamine-D6	142.100	125.100	41.000	13.000	6.000	2.89
CMP	152.163	79.114	41.000	27.000	12.000	4.23
	152.163	77.071	41.000	45.000	0.000	

Trueness of the compound identity was confirmed through comparing the areas of the two MRM transitions, resulting in an identification or ID ratio, also known as an MRM ratio. Every "Q1 Mass" and "Q3 Mass" pairing generated a chromatographic peak. MRM ratios for each compound, with the exception of internal standards, were calculated by dividing the peak area of the second pairing of each compound by the peak area of the first pairing. To build an acceptable ID ratio range, the ratios observed for every calibrator were averaged. For results to be accepted, the ID ratio must be within 20% of the ID ratio average using two significant figures for the percentage value.

Solid Phase Extraction Method

SPE was used to extract and concentrate methamphetamine, pseudoephedrine, CMP, and amphetamine from the wastewater samples. For SPE, the following materials were utilized: Oasis MCX 3 cc (60 mg, 30 μm) cartridges (Waters Corporation, Milford, MA), VacElut 20 Manifold (Agilent Technologies, Santa Clara, CA), CEREX 48 Flow Control and CEREX 48 Sample Concentrator (SPEware Corporation, Baldwin Park, CA). The following solutions were utilized: internal standard Mix (1000 ng/mL solution of all three deuterated internal standards in LC-MS grade water), 10 mM hydrochloric acid (HCl) solution prepared with 37% HCl and LC-MS grade water, LC-MS grade methanol, ACS grade ammonium hydroxide (Fisher Scientific, Hampton NH) and Mobile Phase A. Table 2 outlines each section of the solid phase extraction procedure. Briefly, for every sample, 5 µL of internal standard mix and 20 mL of 10 mM hydrochloric acid solution were added to 50 mL of sample. SPE cartridges were loaded into the CEREX 48 Flow Control and conditioned prior to being moved to the VacElut 20 manifold for sample addition. After sample addition, the cartridges were returned to the CEREX 48 Flow Control for a rinse step and then the cartridges were dried under positive pressure for 20 minutes at approximately 80 psi. After being vacuum dried, elution buffer was added and the eluent was collected into labeled 8 mL plastic test tubes. Samples were dried to complete dryness under nitrogen at 40°C in the CEREX 48 Sample Concentrator. Mobile phase A was used as reconstitution buffer and was added to each test tube. Following thorough vortexing, every sample was transferred to a 1 mL amber LC injection vial.

SPE Step	Parameter
Sample Preparation	50 mL Wastewater Sample
	5 μL Internal Standard Mix
	20 mL 10 mM HCl
Condition	2 mL LC-MS grade methanol
	2 mL 10 mM HCl
	2 mL 10 mM HCl
Sample Addition	50 mL sample
Rinse	2 mL 10 mM HCl
Cartridge Dry Down	20 min at ~80 psi
Elution	2 mL 2% ammonium hydroxide in methanol
Elution Dry Down	Under nitrogen at 40°C
Reconstitution	200 μL Mobile Phase A

 Table 2. Outline of the solid phase extraction procedure.

SPE Method Validation Process

Since the SPE-LC-MS/MS method used for this research was only slightly different from the method developed for the 2016-2017 study, and to save time and resources, a full method validation was not performed. A mini-validation was performed on the calibration model to assess linearity of the calibration curve, as well as the accuracy and precision of each calibrator in the calibration model.

The quantitation ratios, the ratio of the larger transition area to the internal standard transition area, from the calibrators that met the identification criteria were plotted versus concentration. After the data were plotted, they were fitted with a best fit line, and weightings were adjusted to assure the best correlation, or highest R² value. The R² for this line was required to be greater than 0.9. For the calibration points to be included in this study, they had to have an accuracy and precision (%CV) within 20% when applied to the line of best fit. The lower limit of quantitation (LLOQ) was permitted to be within 30% for both accuracy and precision, though its instrument response had to be at least five times greater than the response of a blank. The linear range for all non-internal standard compounds in the LC-MS/MS method mentioned above contained the following calibrator points: 300, 200, 100, 50, and 1 ng/L.

Six replicates of the calibration curve were extracted and concentrations values were calculated for each calibrator. Accuracy for each calibrator was calculated by averaging the concentration of the six replicates and dividing that average by the theoretical concentration of that calibrator. Precision for each calibrator was calculated by dividing the standard deviation average by the calculation average and subtracting that value from 1. R-squared values for each calibration curve were obtained after applying a best fit line, and all values averaged for a given compound. The concentration values, mentioned above, were obtained utilizing MultiQuant software (SCIEX, Foster City, CA), which is specifically designed for LC-MS/MS result analysis. All other values and statistical parameters were obtained by utilizing Microsoft Excel (Microsoft Corporation, Redmond, WA).

Results and Discussion

SPE Validation Results

The following calibrators met criteria of linearity by having accuracy and precision values within 20%: 300, 200, 100, 50, and 1 ng/L. Table 3 through Table 6 demonstrate the accuracy and precision for all calibrator levels of methamphetamine, pseudoephedrine, amphetamine, and CMP. The "Average" column refers to the average concentration, in ng/L, of the 6 replicate runs. Accuracy and precision are reported as percentages, with 100% considered to be absolute. Any value below or above true accuracy or precision is considered a suppression or enhancement of calibrator concentration, respectively. All average concentrations of the blank were under 5 times that of the LLOQ, which was determined to be 1 ng/L for methamphetamine, pseudoephedrine, and amphetamine and 50 ng/L for CMP. In the following sections, any value outside the calibration curve range are estimates based on the slope of each best fit line, but must meet identification criteria to be reported. The best fit line for pseudoephedrine was determined to be a linear fit with $1/x^2$ weighting. The best fit line for pseudoephedrine was determined to be a linear fit with $1/y^2$ weighting.

Table 3. Methamphetamine linearity results.

		Overall	Overall
Methamphetamine Calibrator (ng/L)	Average	Accuracy	Precision
300	296.90	105%	94%
200	202.60	104%	97%
100	109.50	105%	95%
50	40.70	82%	98%
1	1.09	103%	96%
Blank*	0.00		
R ²	0.997		

 Table 4. Pseudoephedrine linearity results.

		Overall	Overall
Pseudoephedrine Calibrator (ng/L)	Average	Accuracy	Precision
300	307.97	103%	97%
200	197.03	99%	99%
100	100.34	100%	99%
50	49.06	98%	94%
1	1.01	101%	92%
Blank*	0.00		
R ²	0.999		

Table 5. Amphetamine linearity results.

		Overall	Overall
Amphetamine Calibrator (ng/L)	Average	Accuracy	Precision
300	304.47	101%	95%
200	202.43	101%	99%
100	100.32	100%	98%
50	50.05	100%	99%
1	1.00	100%	100%
Blank*	0.00		
R ²	0.998		

Table 6. CMP linearity results.

		Overall	Overall
CMP Calibrator (ng/L)	Average	Accuracy	Precision
300	295.53	99%	95%
200	202.10	101%	96%
100	100.96	101%	97%
50	49.55	99%	98%
Blank*	0.00		
R ²	0.995		

Wastewater Analysis

SC-GA Samples

Four shipments of wastewater samples were sent from SC-GA to the OSU-FTTL between May 1, 2017 and December 6, 2017. In total, 73 samples were collected from municipalities in SC and GA and analyzed at the OSU-FTTL. Table 7 summarizes the findings from the SC-GA wastewater samples; for simplicity, both SC and GA samples are collectively referred to as "SC samples" in table enumeration and throughout this report. Analysis of SC 1-18 was conducted May 19, SC 19-28 on June 21, SC 29-46 on October 11 and SC 47-73 on December 6. On May 19, LLOQs were determined to be 1 ng/L for methamphetamine (meth) and pseudoephedrine (pseudo) and 50 ng/L for CMP and amphetamine (amp). On June 21, the LLOQs were determined to be 1 ng/L for pseudo and CMP. On October 11 and December 6, the LLOQs were determined to be 1 ng/L for meth, pseudo, and amp and 50 ng/L for CMP. Concentrations above 300 ng/L exceed the upper limit of quantitation (ULOQ) for the method and are therefore outside of the validated analytical measurement range. These values are estimates based on the equation of a line derived from the calibrators of each drug and are provided for convenience.

Table 7. Concentration of methamphetamine (meth), pseudoephedrine (pseudo), CMP, and amphetamine (amp) observed in wastewater samples collected in select SC-GA municipalities. All concentrations listed in ng/L. Concentrations above 300 ng/L exceed the ULOQ for the method and are estimates provided for convenience.

	Sample Demographics	le Demographics Observed Drug Concentration (r			n (ng/L)	
Sample		grab or				
Number	Collection Site	composite	Meth	Pseudo	СМР	Amp
SC 1	College campus Lift Station (LS)	grab	2	587	0	499
SC 2	Multiple-neighborhood LS	grab	760	0	0	188
SC 3	Neighborhood LS	grab	701	399	0	256
SC 4	Neighborhood LS	grab	22	16	0	1155
SC 5	Multiple-neighborhood LS	grab	2286	465	0	435
SC 6	Neighborhood LS	grab	946	989	0	899
SC 7	Neighborhood LS	grab	23	1219	0	174
SC 8	Multiple-neighborhood LS	grab	26	1482	0	231
SC 9	Neighborhood LS	grab	93	1640	0	244
SC 10	Neighborhood LS	grab	16	2128	0	387
SC 11	Neighborhood LS	grab	26	640	0	678
SC 12	Neighborhood LS	grab	4905	2298	0	729
SC 13	Multiple-neighborhood LS	grab	1426	2062	0	420
SC 14	Neighborhood LS	grab	49	444	0	0
SC 15	Neighborhood LS	grab	0	3158	0	0
SC 16	Neighborhood LS	grab	117	484	0	0
SC 17	Neighborhood LS	grab	0	662	0	253
SC 18	Neighborhood LS	grab	9739	1377	0	1006
SC 19	Multiple-neighborhood LS	grab	295	0	0	140
SC 20	Multiple-neighborhood LS	grab	441	949	0	204
SC 21	Multiple-neighborhood LS	grab	446	762	0	218
SC 22	WWTP inlet feedstream - 30,000 residents	grab	280	345	0	119
SC 23	WWTP inlet feedstream - 20,000 residents	grab	952	1255	0	530
SC 24	WWTP inlet	grab	654	527	0	258
SC 25	WWTP Aeration Basin #1 Weir Outflow	grab	127	80	0	1
SC 26	WWTP Aeration Basin #2 Weir Outflow	grab	0	0	0	0
SC 27	WWTP Secondary Clarifier Outflow	grab	0	0	0	0
SC 28	WWTP Post Chlorine Removal (outfall)	grab	0	0	0	0
SC 29	Detention Center LS Tue	composite	909	94	0	167
SC 30	Detention Center LS Wed	composite	788	0	0	108
SC 31	Detention Center LS Thu	composite	1132	0	0	239
SC 32	Detention Center LS Fri	composite	523	126	0	106
SC 33	Detention Center LS Sat	composite	612	351	0	141
SC 34	Detention Center LS Sun	composite	990	366	0	408

SC 35	Detention Center LS Mon	composite	615	481	0	168
SC 36	Neighborhood LS	composite	712	1568	0	572
SC 37	Neighborhood LS	composite	933	1618	0	647
SC 38	Neighborhood LS	composite	747	954	0	843
SC 39	Neighborhood LS	composite	873	1450	0	498
SC 40	Neighborhood LS	composite	434	1823	0	494
SC 41	WWTP inlet feedstream - 5,000 residents	composite	430	556	0	191
SC 42	WWTP inlet feedstream - 5,000 residents	composite	406	474	0	184
SC 43	WWTP inlet feedstream - 5,000 residents	composite	392	446	0	164
SC 44	WWTP inlet feedstream - 5,000 residents	composite	598	628	0	231
SC 45	WWTP inlet feedstream - 5,000 residents	composite	666	424	0	274
SC 46	WWTP inlet feedstream - 5,000 residents	composite	529	621	0	222
SC 47	Multiple-neighborhood LS - Tue	composite	3447	810	0	643
SC 48	Multiple-neighborhood LS - Thu	composite	1830	1017	0	419
SC 49	Multiple-neighborhood LS - Fri	composite	1265	494	0	287
SC 50	Multiple-neighborhood LS - Sat	composite	1413	1027	0	399
SC 51	Multiple-neighborhood LS	composite	752	391	0	212
SC 52	Neighborhood stormwater manhole - Tue	composite	310	13	0	53
SC 53	Neighborhood stormwater manhole - Wed	composite	181	7	0	30
SC 54	Neighborhood stormwater manhole - Thu	composite	177	16	0	27
SC 55	Neighborhood stormwater manhole - Fri	composite	278	17	0	32
SC 56	Neighborhood stormwater manhole - Sat	composite	183	0	0	28
SC 57	one city block - Wed	composite	9346	28	0	1092
SC 58	one city block - Thu	composite	6497	14	0	99
SC 59	one city block - Fri	composite	5983	1425	0	155
SC 60	one city block - Sat	composite	9604	1570	0	937
SC 61	one city block - Sun	composite	7329	89	0	747
SC 62	one city block - Mon	composite	6044	1863	0	758
SC 63	one city block - Tue	composite	6145	134	0	430
SC 64	one city block - Wed	composite	8601	37	0	939
SC 65	one city block - Thu	composite	5042	90	0	993
SC 66	one city block - Fri	composite	16330	25	0	2692
SC 67	one city block - Sat	composite	16950	34	0	3218
SC 68	one city block - Sun	composite	39980	49	0	7580
SC 69	one city block - Mon	composite	6589	14	0	1337
SC 70	neighborhood manhole	composite	569	1397	0	228
SC 71	neighborhood manhole	composite	650	1544	0	224
SC 72	neighborhood manhole	grab	2437	717	0	327
SC 73	1/2 city block	grab	10150	0	0	201

As can be seen in Table 8, of the 73 samples analyzed from select SC-GA municipalities, 68 (93%) were positive for methamphetamine, 64 (88%) were positive for pseudoephedrine, 0 (0%) were positive for CMP, and 67 (92%) were positive for amphetamine. When comparing the concentration of methamphetamine to the concentration of amphetamine observed in the 73 samples from SC-GA, the data suggests that methamphetamine was of biological origin, as opposed to detection of any dumping of waste from a One Pot methamphetamine lab. When used by humans, methamphetamine has been shown to be excreted as 50% unmetabolized drug and 10-20% as the metabolite amphetamine.⁷ To add to this, amphetamine is not known to be produced in a One Pot methamphetamine lab.⁸ Also, as demonstrated in the 2015-2016 study, if waste from a One Pot methamphetamine lab was collected with one of these wastewater samples, a peak for CMP would likely have been observed, further suggesting the data collected from SC-GA was that of methamphetamine use and not of methamphetamine production.⁶

	Methamphetamine	Pseudoephedrine	CMP	Amphetamine
Number of Positives	68	64	0	67
Percent of Samples Positive	93	88	0	92

Table 8. Total number and percentage of the 73 wastewater samples from SC-GA positive for methamphetamine, pseudoephedrine, CMP, and amphetamine.

Observed methamphetamine and amphetamine spikes seen among the residential lift station grab samples suggest a likely biological origin. Some lift station grab samples presented spikes ranging well above the ULOD of 300 nanograms per liter and appear to range into the nanogram per milliliter level, as seen in samples SC 5, 12, 18, 72 and 73. Oyler et al. reported peak void values above 6,000 micrograms/liter from administered methamphetamine doses of 20mg.⁹ For one 250 mL void, dilution into 500 gallons of sewage water would yield a concentration of 800 nanograms per liter (7,500X dilution), which is consistent with the grab sample data. However, the National Highway Traffic Safety Administration (NHTSA) reported in 2004 methamphetamine "common abused doses are 100-1000 mg/day, and up to 5000 mg/day in chronic binge use."⁷ Our findings of methamphetamine in sewage water at apparent nanogram per milliliter levels is significant in opening the possibility for detection via portable, commercial off-the-shelf instruments with development of simplified field sample preparation steps.

Highest 24-hr composite sample methamphetamine values were seen in SC 66-68, taken from one city block with no sewer feeds from outside this block (headwaters). These values are elevated above the daily samples of the 2 weeks prior and likely indicate weekend binge use from one of the approximately 30 connected residencies. However, the low pseudoephedrine values and lack of CMP each suggest the consumed meth was not of One Pot origin.

WWTP samples showed attenuation of the four monitored chemical compounds as they underwent applied sewage treatment methods. SC 22-24 grab sample inlet values at the large WWTP serving 50,000+ residents and the SC 41-46 composite sample inlet values from the 5,000 resident WWTP both report consistent methamphetamine concentrations of several hundred nanograms per liter. Both methamphetamine and pseudoephedrine grab sample levels in SC 24 at the large WWTP inlet are consistent with a mixture of its two feeder flows, SC 22 and 23. Stepwise attenuation of the monitored chemical compounds by activated sludge microbial processes across the large WWTP is evident when comparing inlet values (SC 24) to reduced Aeration Basin #1 exit weir values (SC 25) and Aeration Basin #2 exit weir zeroing of all target compounds (SC 26).

Deployment of the automated sampler into manhole chimneys, where possible, proved challenging and required some on-the-fly tool adaption to achieve hanging suspension of the sampler and successful inlet tube immersion below the wastewater flow. Exploration of alternatives to achieve reduced setup time behind traffic barricades and easier retrieval included manhole deployment of low cost industrial spill control sorbent pads and a sorbent sock. These sorbent materials were deployed into flowing wastewater and left for several days to investigate simpler sample collection methods, as seen in SC 70 and 71.

In-manhole sampler deployment at a city park into a stormwater access - initially mistaken for a sewer access - proved to yield valuable data seen in SC 52-56. The data suggest a background level of contamination in the low hundreds of nanograms per liter may exist for the neighborhood, arising from surface water runoff from roofs, cars, driveways, streets, etc. and may possibly include dilution by groundwater infiltration as well, as is typical of both sewer and stormwater collection systems.

SC 29-71 evidence multiple-day persistence of target compounds while remaining in undisturbed open collection bottles at ambient temperatures for multiple days, including ng/mL methamphetamine seen in SC 57 following 12 days residence time inside the manhole-deployed sampler prior to retrieval.

Manhole grab sample SC 73, taken mid-block at 10:00 am, presented an estimated methamphetamine concentration of 10,150 ng/L versus the grab sample SC 72, which was taken from a manhole about three blocks downstream 15 minutes later and presented an estimated methamphetamine concentration of 2,437 ng/L. The mid-block large methamphetamine concentration was sampled in gravity feed from about 15 residencies.

Lift Station Headspace VOC Detection

Over a period of 6 days during the field collection of SC 36-39, VOC levels were tracked within the sampled lift station to observe any organic solvents dumped to the sewers from a residence. Figure 10 depicts VOC levels seen by the PID to be at a near-constant, low level

around 5 ppm in the headspace gas of the lift station over the first five days, with one small peak near 60 ppm seen on day six. Photoionization detectors respond to hundreds of organic compounds and some inorganic compounds, including hydrogen sulfide - which may also be the source of corrosion of the USB battery contacts found after the six-day PID residence time in the lift station headspace.



Figure 10. In parallel to the six-day lift station collection of composite wastewater samples SC 36-39, a PID assembly was hung inside the closed lift station headspace to collect data of VOCs present in the sewers. One peak approaching 60 ppm was seen on the final day of data collection.

The unknown compound presenting a peak seen in the PID field data may be any of hundreds of organics detected by ultraviolet lamp excitation within the PID. For comparison, we conducted a laboratory experiment where 250 mL of Crown[®] camp fuel (Packaging Services Company, Pearland, TX) was deposited to a limited-access sewer at 9:50 a.m. and the headspace of the associated lift station was analyzed by a MiniRAE 3000 PGM-7320 handheld PID (RAE Systems, Sunnyvale, CA) for the presence of VOCs. The PID was positioned one foot below the lift station hatch opening, approximately 18 feet above the water line, and the resulting instrument response can be seen in Figure 11. As can be seen, a large spike in VOCs was observed around 10:04 a.m., and was visible until around 10:39 a.m.



Figure 11. The PID response from the headspace of a lift station during a controlled dump of 250 mL of camp fuel into a limitedaccess sewer.

OK Samples

A total of 32 wastewater samples were collected in OK and analyzed at the OSU-FTTL. Table 9 summarizes the findings from the OK wastewater samples. Analysis dates are separated by lines in the table. On July 19 and August 3, the LLOQs for all four compounds were determined to be 50 ng/L. On September 27, the LLOQs were determined to be 1 ng/L for pseudoephedrine ad 50 ng/L for CMP. Due to quantifiable methamphetamine and amphetamine peaks being observed in the sample blanks, the LLOQs for these two drugs were set at 5-times the concentration observed in the blank, making the LLOQ 200 ng/L for methamphetamine and 25 ng/L for amphetamine. Concentrations above 300 ng/L exceed the ULOQ for the method and are therefore outside of the validated analytical measurement range. These values are estimates based on the equation of a line derived from the calibrators of each drug and are provided for convenience. Concentrations denoted with an asterisk were below the LLOQ for the method but met all other criteria for being designated a peak. These values were outside the validated analytical measurement range and are therefore provide for convenience.

Table 9. Concentration of methamphetamine (meth), pseudoephedrine (pseudo), CMP, and amphetamine (amp) observed inwastewater samples collected in OK. All concentrations listed in ng/L. All concentrations listed in ng/L. Concentrations above300 ng/L exceed the ULOQ for the method and are estimates provided for convenience. Concentrations listed with asterisks arebelow the LLOQ but meet all other criteria for being designated a peak.

	Sample Demographics Observed Drug Concentration (ng/L)		(ng/L)			
Sample		Analysis				
Number	Collection Site	Date	Meth	Pseudo	СМР	Amp
OK 1	West Bank (WB) Lift Station	7/19/17	1463	2209	0	209
OK 2	WB Manhole (MH) 1: Western Pines Apt.	7/19/17	3235	364	0	462
OK 3	WB MH2: W. 24th St.	7/19/17	291	0	0	8*
OK 4	WB MH3: Eugene Field Elementary	7/19/17	719	1*	0	61
OK 5	WB MH4: W. 21st St.	7/19/17	1011	58	0	81
OK 6	New Block (NB) Lift Station	7/19/17	4159	759	4*	579
OK 7	NB MH1: Old Jail	7/19/17	1338	0	0	1141
OK 8	NB MH2: Wassco Bottling Co.	7/19/17	4572	489	6*	597
OK 9	NB MH3: Orcutt Machine and Oil Tools	7/19/17	2062	0	0	240
OK 10	NB MH4: S. 38th W. Ave.	7/19/17	6080	2186	17*	1281
OK 11	South Lewis (SL) Lift Station	8/3/17	2388	1990	19*	508
OK 12	SL MH1: Citiplex Towers Parking Lot	8/3/17	860	4310	0	198
OK 13	SL MH2:	8/3/17	183	246	0	1093
OK 14	SL MH3: Deerfield Estates Apt.	8/3/17	0	4560	0	0
OK 15	SL MH4:	8/3/17	0	28	0	0
OK 16	SL MH5	8/3/17	2814	2602	0	632
OK 17	SL MH6: Wal-Mart Parking Lot	8/3/17	136	0	0	0
OK 18	SL MH7: River Spirit Casino Hotel	8/3/17	2388	74	13*	864
OK 19	Clark Park	9/27/17	10*	0	0	5*
OK 20	117th E. Pl and 2nd St. S.	9/27/17	1459	0	0	89
OK 21	Across from Continental Carbonic Products	9/27/17	2354	373	0	221
OK 22	S. 122nd E. Ave and E. 4th Pl. S.	9/27/17	4151	2936	0	489
OK 23	Aspen Manufactured Homes	9/27/17	10040	1886	0	5493
OK 24	Knights Inn	9/27/17	4109	220	0	1201
OK 25	Daylight Donut Flour Co.	9/27/17	782	162	0	168
OK 26	Ridgeview Apt.	9/27/17	1346	0	0	43
OK 27	Mingo Creek across from Meadowbrook Apt.	9/27/17	3475	1380	0	710
OK 28	Mingo Creek across from E. 7th St. S.	9/27/17	431	405	0	115
OK 29	S. 103rd E. Ave between 12th St. and 14th St.	9/27/17	8873	113	0	2505
OK 30	S. 105th E. Ave between 12th St. and 14th St.	9/27/17	1391	160	0	424
OK 31	Greenleaf Wholesale Flowers	9/27/17	3691	142	0	432
OK 32	Sierra Pointe Apt.	9/27/17	1108	11	0	230

As can be seen in Table 10, of the 32 samples analyzed from OK, 30 (94%) were positive for methamphetamine, 25 (78%) were positive for pseudoephedrine, 5 (16%) were positive for CMP, and 29 (91%) were positive for amphetamine. When comparing the concentration of methamphetamine to the concentration of amphetamine observed in the 32 samples from OK, the data suggests that methamphetamine use, as opposed to the dumping of waste from a One Pot methamphetamine lab, was observed in all but 5 of the wastewater samples. The 27 samples that are suggestive of methamphetamine use contained the methamphetamine metabolite amphetamine and contained no measurable CMP, suggesting that no One Pot methamphetamine lab waste was present in these samples. However, the remaining 5 samples analyzed from OK contained CMP peaks. All CMP peaks were below the LLOQ of 50 ng/L, but they met all other criteria for being designated a positive peak. While it has been shown that CMP is at least partially excreted by humans as unmetabolized drug, of 103 urine samples previously reported positive for methamphetamine analyzed at the OSU-FTTL, only 1 tested positive for CMP, suggesting CMP is present in larger amounts in One Pot methamphetamine waste than in human urine.¹⁰ The very low CMP levels detected in this study may be suggestive of either the presence of waste from a One Pot methamphetamine lab or possibly CMP excreted in urine; as all values found were below LLOQ, source discrimination is challenging. In either case, we assess the found CMP as signatory of One Pot meth lab origin.

Table 10. Total number and percentage of the 32 wastewater samples from OK positive for methamphetam	nine,
pseudoephedrine, CMP, and amphetamine.	

	Methamphetamine	Pseudoephedrine	CMP*	Amphetamine
Number of Positives	30	25	5	29
Percent of Samples	94	78	16	91

*All CMP positives were below the LLOQ of 50 ng/L but met all other criteria to be designated a positive peak.

The first and second set of samples collected in OK represents a snapshot of the communities sampled. These 18 samples were randomly taken from easily accessible manhole covers and wastewater lift stations around OK to get an idea of the methamphetamine, pseudoephedrine, CMP, and amphetamine levels in the wastewater collection system. As can be seen in Table 9, methamphetamine, pseudoephedrine and amphetamine were observed in most of the collected samples. CMP was observed in 5 of the 18 samples, though the concentration of CMP was below the LLOQ of 50 ng/L in each of these 5 samples, so these samples are only suggestive of a CMP positive. Amphetamine was present in every sample that contained methamphetamine, but was not observed in cook waste from One Pot production in the FY16 study, creating a useful marker in sewage water. Presence of amphetamine may suggest prescription use, illicit use, or metabolized methamphetamine. Unlike the SC-GA samples, several of the OK samples had suggestive CMP positives, which invites the possibility that some of the methamphetamine and pseudoephedrine observed may be from One Pot users or the waste of a One Pot methamphetamine and pseudoephedrine observed may be definitively determined.

The third set of samples collected, OK 19-32, were from areas that local law enforcement has historically found larger numbers of One Pot methamphetamine labs when compared to other areas of OK. As can be seen in Table 9, all the samples collected from these areas were positive for methamphetamine and amphetamine, and all but one was positive for pseudoephedrine. None of the samples collected during the third OK collection were positive for CMP, suggesting the methamphetamine observed in the samples was from methamphetamine use and not from One Pot waste. Two samples collected during the third OK collection showed the potential of using this technique as a way of identifying residencies where methamphetamine is being used. OK 29 and OK 30 were collected from consecutive manhole locations along the same wastewater gravity main. OK 29 had an additional 34 houses feeding the gravity line as compared to OK 30. OK 29 contained six times the concentration of methamphetamine and amphetamine and amphetamine user among the 34 residencies upstream of the manhole where OK 29 was collected.

While the goal of this study was to identify locations where methamphetamine is being produced via the One Pot, declining domestic methamphetamine production has made this goal difficult to achieve.² However, the ability of this method to identify locations where methamphetamine is being used may still be of importance for law enforcement agencies.

Conclusion

In summary, 105 wastewater samples were collected and analyzed for the presence of methamphetamine, pseudoephedrine, CMP, and amphetamine. Of the 73 samples collected in SC-GA, none were positive for CMP. Of the 32 samples collected in OK, 5 were positive for CMP, though the concentration of CMP in these samples were below the LLOQ of 50 ng/L. Results from the controlled deposit to sewer of One Pot cook waste upstream of a sampled lift station in FY16 indicated a tandem spike of methamphetamine and pseudoephedrine at ng/mL levels may be seen over a two-hour period following such a deposit. New data in FY17 from sewage water collection systems in SC, GA and OK indicate levels of methamphetamine and pseudoephedrine in the range of nanograms per milliliter occur routinely in the municipality collection systems sampled and would likely or almost certainly obfuscate use of these 2 targets as indicators of cook waste deposited to sewer. The new data suggests nanogram per milliliter levels of methamphetamine may well occur routinely as a biological marker arising from users – further supported by the robust presence of known metabolite amphetamine found alongside the higher values for methamphetamine. However, nanogram per milliliter presence of pseudoephedrine is likely to be from over-the-counter consumption of pseudoephedrine. By contrast and interestingly, the highest levels of methamphetamine in this study correspond to very low pseudoephedrine levels and is therefore suggestive the high consumption was not sourced from a One Pot lab. These highest levels of meth seen in the one-block sample collection occurred over a weekend, very suggestive of weekend binge use at this location. The lack of correlation between pseudoephedrine and methamphetamine levels seen in early field samples disproved the starting hypothesis of a tandem spike of methamphetamine and pseudoephedrine seen together in sewage water to be useful as a unique signature of One Pot lab waste. The starting hypothesis was inferred from FY16 data observed following controlled One Pot lab waste deposits to sewer in Year 1 of this work. Following frequent discovery of high levels of both meth and pseudo in early sample collections, CMP was pursued as a potential indicator of One Pot methamphetamine, either sourced from urine as an unmetabolized, trace byproduct found in consumed One Pot meth, or originating from One Pot lab waste dumped to sewer.

The low number of CMP positives, and thus assumed One Pot methamphetamine labs, observed in this study can perhaps be explained by the current trends in methamphetamine use and production. According to the DEA, methamphetamine use is on the rise, due to the high availability of methamphetamine coming into the United States from Mexico and the record low prices of the drug.² Because of the current ease in obtaining low-cost methamphetamine from Mexico, many methamphetamine users have switched from producing their own methamphetamine to purchasing from dealers that have had the drug smuggled the drug into the United States by the Mexican cartels. The current influx of cheap methamphetamine, alongside tighter state regulations on methamphetamine precursors, has led to a 16-year low in domestic methamphetamine production.²

While current domestic production of methamphetamine is low, intelligence suggests that Mexican cartels are beginning to focus their efforts on methamphetamine distribution down

the east coast of the United States.² It is the belief of the DEA that as new customers begin using methamphetamine, the price will begin to rise and this rise in methamphetamine price will lead to more people once again producing methamphetamine.² If domestic methamphetamine production increases, this research could prove to be beneficial in the identification of clandestine One Pot methamphetamine labs. Currently, this research has shown that wastewater can be used to identify neighborhood-level, street-level, and binge use of methamphetamine. Sewage water testing for CMP at very trace levels was found to be a possible indicator of fixed localities with One Pot methamphetamine consumption by users or production by cooks. Further, trace analysis for pseudoephedrine and amphetamine levels was found to be useful as a signature to demonstrate the lack of a One Pot meth lab where high methamphetamine use was observed in a one-block street, with sourcing narrowed to as few as 15 residencies.

References

- Gonzales R, Mooney L, Rawson RA. The Methamphetamine Problem in the United States. Annu Rev Public Health. 2010;31(1):385-398. doi:10.1146/annurev.publhealth.012809.103600
- 2. 2017 National Drug Threat Assessment. U.S. Department of Justice Drug Enforcement Administration; 2017:67-82. https://www.dea.gov/docs/DIR-040-17_2017-NDTA.pdf.
- 3. Person EC, Meyer JA, Vyvyan JR. Structural determination of the principal byproduct of the lithium-ammonia reduction method of methamphetamine manufacture. *J Forensic Sci*. 2005;50(1):1-9. doi:10.1520/JFS2003389
- Wastewater treatment (% population connected). OECD Web site. http://stats.oecd.org/index.aspx?DataSetCode=water_treat. Published 2015. Accessed December 21, 2017.
- 5. *People v. Electronic Plating Company*.(Appellate Court of Illinois, First District, Fourth Division 1997). http://caselaw.findlaw.com/il-court-of-appeals/1076769.html. Accessed December 21, 2017.
- 6. Wagner J, Green M, Ciesielski A, Pretorius D. *Detection of One Pot Methamphetamine Production Effluents in Waste Water*. Washington, DC: NIJ
- 7. Couper FJ, Logan BK. *Drugs and Human Performance Fact Sheets*. Washington, DC: National Highway Traffic Safety Administration; 2004:61-66.
- 8. Ciesielski A. Characterization of clandestine One Pot methamphetamine laboratories using gas chromatography-mass spectrometry and liquid chromatography-tandem mass spectrometry. May 2017.
- 9. Oyler JM, Cone EJ, Joseph RE, Moolchan ET, Huestis MA. Duration of Detectable Methamphetamine and Amphetamine Excretion in Urine after Controlled Oral Administration of Methamphetamine to Humans. *Clin Chem.* 2002;48(10):1703-1714.
- 10. Green MK. Environmental impacts of one pot methamphetamine laboratories characterization and detection of trace materials. May 2017.