Document Title:	**One Pot Methamphetamine Laboratory Ambient Standoff Detection and Decontamination**
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Document Number:	**254673**
Date Received:	**April 2020**
Award Number:	**IAA 2016-DNR-5470 (DOE SRNL)**

One Pot Methamphetamine Laboratory Ambient Standoff Detection and Decontamination

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Executive Summary

Seven One Pot methamphetamine lab cooks were performed at the Oklahoma State University Fire Research and Training Center in an open-sided plastic garden shed to simulate a residential cook location. Engine starting fluid and camp fuel were alternated as the solvents used for the One Pot cooks. Decontamination was performed between each cook by spraying down the SCBA air tanks and PPE of the researchers and at the end of each day by spraying down the shed interior surfaces as well. Contamination levels observed before and after each cook were tested using lateral flow immunoassays, and were later quantified by laboratory analysis of split samples. Active air samplers with PTFE filters were employed to sample airborne particulates within the cook shed; passive air sampling was achieved using surface-treated vacuum canisters, body-worn helium diffusion sampling personal monitors and diffusive sorbent pens packed with organic sorbents, and open vial grab sampling. Following the final cook, the spent One Pots were collectively burned to visually observe the performance of several fire prevention or extinguishing methods currently recommended for controlling One Pot meth lab fires.

Effluents identified in shed ambient air include ammonia and numerous VOCs, including straight-chain hydrocarbons, aromatic and substituted aromatic hydrocarbons, ethyl ether, and methamphetamine. Wipe samples showed methamphetamine contamination levels ranged between 0.10 and 264.35 ng/100 cm^2. A mobile vehicle equipped with instrumentation meant to aid in industrial gas leak detection and repair proved effective in detecting ammonia and the presence of VOCs at standoff distances using an on-board differential ultraviolet absorption spectrometer (DUVAS) and both an on-board photo ionization detector (PID) and remote PIDs placed at distances up to 100 meters from the cook shed. A portable integrated cavity output spectrometer detected ammonia plumes at up to 100 meters and discriminated controlled cook "burp" releases at 40 meters. Wind direction was a primary factor noted in detection capability of remote instrumentation.

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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 allows for methamphetamine production to occur in 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 these electrons 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. The ammonia gas bubbles through an organic solvent to reach the lithium metal and strip it of electrons, which are then used to reduce the pseudoephedrine or ephedrine to methamphetamine.

Another gas generated during the One Pot production of methamphetamine, or a "cook", is hydrogen chloride (HCl) gas. HCl gas is bubbled into the organic solvent after the cook has come to completion, causing the methamphetamine to precipitate out of the organic solvent as a hydrochloride salt so it can be recovered in a usable form. Both ammonia gas and HCl gas are classified as corrosive gases that pose a significant health hazard to people who are exposed to them without the proper personal protective equipment (PPE).[4,5] To add to this health hazard, the organic solvents used during a One Pot cook are volatile, causing them to readily become a gas that can be inhaled and cause respiratory issues in people within and near clandestine meth labs. Methamphetamine itself can also be released in a gaseous state during a One Pot methamphetamine cook, adding yet another respiratory hazard to those who are within close proximity of a One Pot cook.[6]

This study utilized these gases that are released into the environment by One Pot methamphetamine cooks as a potential way to identify locations were such cooks are taking place. Several previous studies have been performed to determine what type of gases are present during other routes of methamphetamine production, but here we present the first cook plume detection study of the One Pot route of methamphetamine production.[7,8] For this study, One Pot methamphetamine cooks were performed in a garden shed to simulate the environment that methamphetamine may be produced in these small-yield clandestine labs. Air monitoring was performed inside the garden shed, as well as from varying distance downwind of the cook site to identify gases released during a One Pot methamphetamine cook and concentrations present. Between cooks, the garden shed was decontaminated following the guidelines developed by the United States Environmental Protection Agency (EPA) and swab sampling was performed to determine the effectiveness of this mode of meth lab decontamination.[9] The goal of this research was to: a) determine the chemical exposure level of people living in a residence where methamphetamine is/was produced via a One Pot b) determine if the gases being generated by a One Pot methamphetamine could be detected with stand-off instrumentation c) determine if the decontamination process recommended by the EPA for remediation of former methamphetamine laboratories is successful in reducing meth lab-related chemical contamination to a safe level.

Materials and Methods

One Pot Cooks

Six One Pot methamphetamine cooks were scheduled to be performed from Tuesday, November 28, 2017 through Thursday, November 30, 2017 at the Oklahoma State University Fire Research and Training Center in Stillwater, OK. The 6 One Pot methamphetamine cooks performed were divided between 3 using starting fluid (ethyl ether) as an organic solvent and 3 using camp fuel (light-medium petroleum distillate) as an organic solvent. As seen in Figure 1, the One Pot methamphetamine cooks were performed in a plastic garden shed using a modified version of a recipe known to be used by methamphetamine producers in order to best mimic the environment such a cook may be performed in.[10] For safety, researchers performing the cooks were dressed in level B protection (See Figure 2), including chemically and thermally protective suits and self-contained breathing apparatuses (SCBA). As seen in Figure 3, a firefighter was on standby in turnout gear and a SCBA with a primed water hose to prevent fire-related injuries and minimize damage to the laboratory setup.

Figure 1. The set up for the One Pot methamphetamine cooks performed in this study. Cooks were performed in a plastic garden shed to simulate what a real cook environment may be like.

Figure 2. Level B protective suit, including chemically and thermally protective suits and a self-contained breathing apparatus.

Figure 3. To mitigate fire hazards associated with One Pot methamphetamine labs, a trained firefighter was on standby in turnout gear with a primed hose throughout the duration of the cooks.

For the One Pots, 600 mg of ground pseudoephedrine was added to a clean, 32 oz plastic bottle. The contents of a 6”x9” instant cold compress pack (GoGoods.com, Inc, Columbia, MD) (ammonium nitrate) were then added to the plastic bottle, followed by either 2.5 cans of Prestone® Premium Starting Fluid (Prestone Products Corporation, Chicago, IL) or 600 mL of Coleman camp fuel (Coleman Company, Wichita, KS). Before the starting fluid could be added to the plastic bottle, the cans needed to be depressurized. This was done by inverting the cans while holding down the dispenser button until air no longer expelled. After the addition of the organic solvent, a single capful of Drain Out Crystal Clog Remover (Summit Brands, Fort Wayne, IN) (sodium hydroxide) (See Figure 4) was added to the cook bottle, followed by approximately 6 mL of water to catalyze the production of ammonia gas. Six 0.5 g strips of lithium ribbon (Sigma-Aldrich Corp, St. Louis, MO) were then added to the plastic bottle and the bottle was capped.

Figure 4. Sodium hydroxide was added to the One Pot methamphetamine cook in capful aliquots as deemed necessary for each individual reaction.

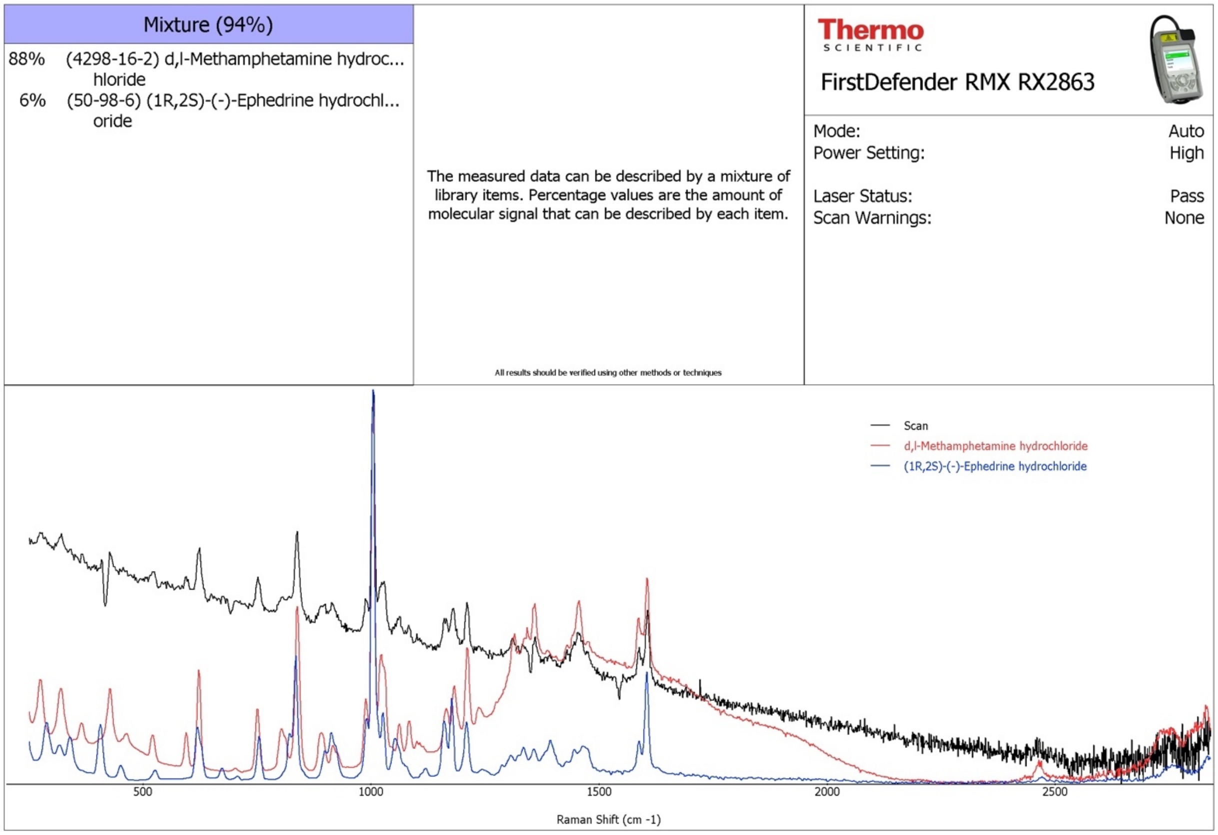
Once the bottle was capped, it was swirled to allow the water that was added to the bottle to interact with the ammonium nitrate and the sodium hydroxide (Drain Out), generating ammonia gas. The One Pot was placed in a ring stand to prevent tipping and was allowed to react, or “roll”, for 1 hour. Every 5 minutes, the lid to the bottle was opened slightly to “burp” the reaction, allowing some ammonia gas to be released and thus relieve the pressure within the bottle. If the reaction slowed and the rolling ceased, another capful of Drain Out was added to the bottle during the next burping step.

After 1 hour of rolling, the cap was slowly removed from the bottle, fully releasing the ammonia gas from the bottle. Using forceps, the lithium strips were removed from the One Pot and placed under Equate mineral oil (Wal-Mart Stores, Inc, Bentonville, AR) to mitigate flammability. The solvent from the One Pot was then poured through two coffee filters (Farmer Bros Co, Ft. Worth, TX) into a clean, one-pint Mason jar (Kerr Glass Manufacturing Corp, Lancaster, PA). The plastic cook bottle was then rinsed with an additional 200 mL of organic solvent, and that additional solvent was also poured through the coffee filters into the Mason jar. After the organic solvent had been filtered into the Mason jar, one inch of Great Value iodized salt (Wal-Mart Stores, Inc, Bentonville, AR) was added to a hydrogen chloride gas generator, comprised of a clean, 20 oz plastic bottle with a hose protruding from the cap (See Figure 5). Approximately 9 mL of Rooto Professional Drain Opener (Rooto Corporation, Howell, MI) (sulfuric acid) was added to the bottle and the cap was quickly screwed on. The salt-sulfuric acid mixture generated HCl gas, which was bubbled into the filtered organic solvent from the One Pot, causing a powder to precipitate out of the solvent.

Figure 5. A typical hydrogen chloride gas generator, comprised of a plastic bottle with a hole in the cap and a hose protruding from the hole.

Following precipitation, the organic solvent and powder mixture was separated via vacuum filtration. The powder was then air dried at ambient conditions to allow for any remaining solvent to evaporate from the powder. The resulting powder was subjected to a NIK Public Safety Narcotics Identification System presumptive colorimetric test (NIK Public Safety Inc, Jacksonville, FL) as well as Raman spectrometry to demonstrate successful conversion of pseudoephedrine to methamphetamine. The NIK colorimetric test used was Test U: Methamphetamine or MDMA (Ecstasy), which turns dark purple when methamphetamine is present (See Figure 6). The Raman spectrometer used for field identification of methamphetamine was a FirstDefender RMX RX2863 Raman spectrometer (Thermo Scientific, Waltham, MA) and a sample spectrum obtained from this instrument can be seen in Figure 7. Once the production of methamphetamine had been confirmed, all of the One Pot lab, including the liquid waste, solid waste, and the powder were disposed of in a manner deemed suitable by the Oklahoma Bureau of Narcotics and Dangerous Drugs (OBNDD).

Figure 6. An example of a positive NIK Public Safety Narcotics Identification System presumptive colorimetric test. Test U: Methamphetamine and MDMA was used to identify the presence of methamphetamine in the salts produced by each One Pot.



Mixture (94%)	
88%	(4298-16-2) d,l-Methamphetamine hydroc... hloride
6%	(50-98-6) (1R,2S)-(-)-Ephedrine hydrochl... oride

The measured data can be described by a mixture of library items. Percentage values are the amount of molecular signal that can be described by each item.

All results should be verified using other methods or techniques

Thermo SCIENTIFIC

FirstDefender RMX RX2863

Mode:	Auto
Power Setting:	High
Laser Status:	Pass
Scan Warnings:	None

Figure 7. Raman spectrum obtained from analysis of salts produced during a One Pot methamphetamine cook; red and blue spectra display library search results matching the obtained sample spectrum.

Surface Contamination Sampling

To determine the level of surface contamination that was present due to the One Pot methamphetamine labs, eleven locations within the cook shed, two locations directly outside the cook shed, and the arms, legs, chest, masks, and air tanks of the researcher's PPE were sampled prior to the One Pot cooks, immediately following the One Pot cook, and then after a wet decontamination procedure (See Figures 8 and 9). Sampling locations measured 10 x 10 cm (100 cm^2) and were swabbed using a sterile cotton swab wetted with phosphate-buffered saline (PBS) with Triton^{TM} X-100 added as a surfactant. The sampled areas were first swabbed top-to-bottom, then left-to-right, and lastly top-to-bottom a second time to ensure the entire area was thoroughly sampled for trace particulates. Gloves were changed between each sampling event to avoid cross contamination. The wet decontamination procedure used during this study consisted of hosing off the protective suits worn by the researchers performing the One Pot methamphetamine cooks, as well as the cook shed interior, with a copious water stream from the fire hose that was present during the cooks for fire safety. Decontamination was performed on the PPE of the researchers after each cook and on the cook shed at the end of each day.

Figure 8. Three of eleven 10 x 10 cm (100 cm^2) sampling locations selected within the One Pot methamphetamine cook shed.

Figure 9. Surface contamination sampling of one of the researchers prior to beginning a cook.

After wiping the defined sample area, the swab was placed back into a vial with PBS-Triton^{TM} X-100 and allowed to sit. After several minutes, three drops of the drug-PBS-Triton^{TM} X-100 solution were removed from the vial with a 0.5 mL transfer pipette and added to a lateral flow immunoassay (LFIA) cassette developed by the Division of Applied Research and Technology at the Centers for Disease Control and Prevention's National Institute for Occupational Safety and Health (CDC-NIOSH, Cincinnati, OH).[11] The LFIA is a competitive inhibitory immunoassay with a built-in negative control to ensure the assay is functioning properly. After addition of the drug-PBS-Triton^{TM} X-100 solution, the sample migrated via capillary action toward immobilized antigens developed to bind to methamphetamine. If methamphetamine is present in the sample it will preferentially bind to the antigens, causing the colorimetric indicator to be displaced and displaying only the red line from the negative control in the visible results section of the cassette. If methamphetamine is not detected, the colorimetric indicator is not displaced and two red lines appear in the test results section of the immunoassay cassette (See Figure 10).

Figure 10. Competitive lateral flow immunoassay cassette developed by CDC-NIOSH for detection of methamphetamine at a concentration of 50 ng/100 cm^2. Sample 1A was taken pre-cook and sample 1B was taken from the same location post-cook. Two red lines indicate the absence of methamphetamine in the sample. One red line indicates the presence of methamphetamine in the sample.

The LFIAs were developed by CDC-NIOSH as a qualitative method of methamphetamine detection for levels of methamphetamine that exceed 50 ng/100 cm^2. To quantitative the level of methamphetamine contamination from the interior surfaces of the cook shed and the researcher's PPE, the remaining drug-PBS-Triton^{TM} X-100 solution from the LFIAs was sent to the CDC-NIOSH Taft Laboratory in Cincinnati, Ohio, for analysis using a fluorescence covalent microbead immunosorbent assay (FCMIA) developed by the Luminex Corporation (Luminex Corporation, Austin, TX).[12,13] The method used to analyze the samples with FCMIA was first developed by Smith et al in 2010 and has a quantitative range of 0-15 ng/mL for methamphetamine.[14] Briefly, methamphetamine calibrators were prepared at 15, 7.5, 3.75, 1.88, 0.94, 0.46, 0.23, and 0 ng/mL in PBS-Triton^{TM} X-100 diluted 1/3 with storage/blocking buffer (PBS, 1% Bovine serum Albumin (BSA), 0.05% sodium azide, pH=7.4). Fifty microliters of methamphetamine conjugated microspheres at a concentration of 1x10^5 microspheres/mL in storage/blocking buffer were added to the wells of a 1.2 µm filter membrane microtiter plate (Merck Millipore Co, Burlington, MA) and the liquid was aspirated via a Millipore vacuum manifold. After the wells were dried, 50 µL of the calibrators or drug-PSB-Triton^{TM} X-100 solutions were added to the wells, along with 50 µL of primary anti-methamphetamine antibodies at a 1/250,000 dilution in storage/blocking buffer. The microspheres, primary antibodies, and samples were then allowed to incubate at 37^{o}C for 30 minutes on a microplate shaker in the absence of light.

After incubation, the wells of the microtiter plate were washed three times with wash buffer (PBS, 138 mM sodium chloride, 2.7 mM potassium chloride, 0.05% Tween® 20). 50 µL of 5 µg/mL biotin labeled, anti-mouse IgG in storage/blocking buffer was added to the wells, and the plate was again allowed to incubate at 37^{o}C for 30 minutes on a microplate shaker in the absence of light. Following the second incubation, the wells were once again washed three times with wash buffer and then 50 µL of 4µg/mL streptavidin R-PE reporter in storage/blocking buffer was added to the wells. The plates were then incubated a third time at 37°C for 30 minutes on a microplate shaker in the absence of light.

Following the final incubation, the wells were washed three times with wash buffer and the microspheres were resuspended in 100 µL of was buffer. The microtiter plate was then shaken vigorously for 1 minute to disperse the microspheres and the plate was loaded onto the autosampler of the LUMINEX 100 instrument. The LUMINEX 100 was programmed to collect data from 100 microspheres per sample and report the median fluorescence intensity (MFI) of the microsphere-drug conjugate-primary anti-drug conjugate IgG antibody-secondary anti-IgG-biotin-avidin complex. Since the FCMIA is a competitive immunoassay, the more methamphetamine present in the sample loaded into the microtiter plate well, the less the sample fluoresced.

Shed Interior Air Monitoring

Airborne particulate trapping and bulk air sampling was performed inside the cook shed before, during and after the One Pot methamphetamine cooks. Air samples were collected using battery-powered, active air samplers provided by CDC-NIOSH as well as vacuum canister-style passive air samplers provided by Entech Instruments (Entech Instruments, Simi Valley, CA). The active air samplers were utilized for all three ether One Pots and the last two camp fuel One Pots. The passive air samplers were utilized for the first two ether One Pots and the first two camp fuel One Pots.

Active Air Sampling

Prior beginning the One Pot methamphetamine cooks, the five SKC Airchek 2000 active air samplers (SKC Inc, Eighty Four, PA) were each fitted with a 37 mm x 2 µm PTFE filter and the pumps were set to draw in air at a rate of 1.5 L/min. The PTFE filters were used to trap particle phase drugs found in the air that was pulled through the active air samplers. Four of the active samplers were set up inside the shed in two locations while the fifth sampler was fitted to one of the researchers performing the One Pot methamphetamine cook (See Figure 11). As shown in Figure 12, the two locations housing the other four samplers were to the left of active One Pot and behind and to the right of the researchers. The active air sampler fitted to the researcher and one active air sampler from each location within the shed began pulling air through the filter immediately prior to starting a One Pot cook. The remaining two active air samplers, one from each location within the shed, began pulling air through the filter after the One Pot was filtered, just prior to assembly of the HCl gas generator. Two PTFE filters were treated as field blanks; one was briefly exposed to the environment near the shed before any One Pot methamphetamine cooks were performed and the second was exposed to the environment near the shed 30 minutes after the last One Pot methamphetamine cook had concluded. During the final ether One Pot methamphetamine cook, the pumps containing filters 25 and 26 were not turned on, resulting in no data collection from these filters.

Additionally, the pumps containing filters 7 and 8 were actively sampling during the first camp fuel bottle failure. These pumps remained on throughout the duration of the cook that resulted in the bottle failure, as well as the second camp fuel cook, which was performed immediately following the bottle failure. Table 1 summarizes the sampler location, the time each active sample pump was on, and the average flow rate of the air being pulled through the sampler.

Figure 11. An active air sampler fitted to a researcher as they performed a One Pot methamphetamine cook.

Figure 12. Locations of 4 out of 5 active air samplers. Two samplers were placed in each location designated by the orange circles. Two samplers were located left of the active One Pot methamphetamine lab and two samplers were located behind and to the right of the researchers performing the One Pot methamphetamine cooks.

Table 1. Active air pump locations and volumetric sampling information.

	Filter Number	Pump S/N	Start Time	End Time	Pump Run Time (min)	Avg. Total Flow Rate (mL/min)	Total Volume Sampled (L)	Pump Location	Activated Period	Cook Type
Cook 2	1	59755	14:25	16:42	140	1490.25	210.00	Front Left	Full	Camp Fuel
	2	50876	14:26	16:42	135	1483.85	200.70	Back Right	Full	Camp Fuel
	3	34665	14:23	16:41	138	1486.95	206.20	Researcher	Full	Camp Fuel
	4	34643	16:00	16:42	37	1482.00	68.00	Front Left	Salting Out	Camp Fuel
	5	34747	16:01	16:42	37	1512.65	56.00	Back Right	Salting Out	Camp Fuel
	6	-	-	-	-	-	-	Field Blank	Flash Exposure	Field Blank
Cook 3	7	59755	9:22	10:43	71	1510.50	107.25	Front Left	Full	Camp Fuel
	8	50876	9:23	10:43	70	1498.60	104.90	Back Right	Full	Camp Fuel
	9	34665	9:23	10:22	58	1507.40	87.43	Researcher	Full	Camp Fuel
	10	34643	12:20	12:32	14	1450.65	20.31	Front Left	Salting Out	Camp Fuel
	11	34747	12:14	12:32	18	1496.05	26.93	Back Right	Salting Out	Camp Fuel
Cook 4	12	34665	14:17	16:11	114	1506.15	171.70	Researcher	Full	Ether
	13	59755	14:15	16:08	111	1511.80	167.81	Front Left	Full	Ether
	14	50876	14:15	16:08	111	1506.55	167.23	Back Right	Full	Ether
	15	34747	15:41	16:08	31	1629.55	50.52	Back Right	Salting Out	Ether
	16	34643	15:41	16:06	29	1492.85	43.00	Front Left	Salting Out	Ether
Cook 5	17	59755	9:26	11:16	104	1508.90	156.93	Back Right	Full	Camp Fuel
	18	50876	9:26	11:16	104	1513.55	157.41	Front Left	Full	Camp Fuel
	19	34665	9:24	11:17	113	1404.70	158.73	Researcher	Full	Camp Fuel
	20	34643	10:58	11:16	13	1499.70	19.50	Back Right	Salting Out	Camp Fuel
	21	34747	10:58	11:16	13	1629.20	21.18	Front Left	Salting Out	Camp Fuel
Cook 6	22	59755	13:04	14:38	94	1491.05	140.15	Front Left	Full	Ether
	23	50876	13:04	14:38	94	1490.35	140.09	Back Right	Full	Ether
	24	34665	13:04	13:13	9	1226.10	11.03	Researcher	Full	Ether
	25	34643	-	-	-	-	-	Front Left	-	Ether
	26	34747	-	-	-	-	-	Back Right	-	Ether
	27	-	-	-	-	-	-	Field Blank	Flash Exposure	Field Blank

Upon completion of the One Pot methamphetamine cooks, the PTFE filters were sent to the CDC-NIOSH Taft Laboratory in Cincinnati, OH for quantitative analysis of the contaminates collected from the air samples. At CDC-NIOSH, drugs trapped on the PTFE filters were extracted with PBS-Triton^{TM} X-100 and then analyzed using the same FCMIA method as previously described for the surface swab samples.

Passive Air Sampling

Passive air sampling was achieved using vacuum canister-style samplers provided by Entech Instruments. Ten air samplers were utilized for each of the four One Pot methamphetamine cooks monitored. The 10 samplers utilized included 3 Silonite^{TM} treated, 1 L vacuum MiniCans^{TM}; 2 helium diffusion sampling (HDS) personal monitors clipped to the upper-left strap of the SCBA air tank harness; 4 Diffusive Sorbent Pens (DSP), 2 packed with Carbo Pack X and 2 packed with Tenax, clipped to the upper-right strap of the SCBA air tank harness; and one

40 mL screw-top vial grab sampler. Each researcher performing the One Pot methamphetamine cooks had a HDS sampler and set of DSP samplers, one packed with Carbo Pack X and one packed with Tenax. An example of the passive samplers used during the shed interior air monitoring study can be seen in Figure 13.

Figure 13. Passive air monitors provided by Entech Instruments for the shed interior air monitoring study. a.) 1L Minican^{TM} vacuum canister, b.) Helium diffusion sampler (HDS) personal monitor, c.) A set of diffusive sorbent pens (DSPs), one packed with Carbo Pack X and one packed with Tenax, and d.) a 40 mL screw-top grab sampler.

The 3 samplers clipped to each researcher passively collected air samples throughout the duration of each cook. The 1 L vacuum canister grab samples were collected at 3 points throughout the One Pot methamphetamine cooks: prior to the start of the cooks, after the cooks were complete but before salting out began, and after the powdered methamphetamine had been separated from the post-salt solvent via filtration. The 40 mL screw-top vial grab sampler was left open to the environment beginning just prior to assembling the HCl gas generator and was capped following filtration of the powdered methamphetamine from the post-salt solvent. Table 2 summarizes the sampler demographics and the time each sampler was active.

Upon completion of the One Pot methamphetamine cooks, all of the passive air samplers were sent to Entech Instruments for quantitative, semi-quantitative, and qualitative analysis of the contaminates collected from the air samplers. The 1 L MiniCans^{TM} and the HDS personal monitors were analyzed using an Entech 7200 preconcentrator and 7650-M autosampler coupled to an Agilent 6890 gas chromatography instrument with an Agilent 5973 mass spectrometer (Agilent Technologies, Santa Clara, CA). This instrumental set up is shown in Figure 14. The Entech 7200 preconcentrator was set up to concentrate 100 mL of the air samples from the 1 L MiniCans^{TM} to a volume of 1 µL, compressing the volume of the MiniCans^{TM} by 100,000 fold and it concentrated the entire air samples from the HDS personal monitors from a volume of 16 mL to 10 mL, compressing the volume of the HDS personal monitors by 1.6 fold.

Table 2. Passive air sampler demographics and sample collection time.

	Sampler Type	Serial Number#	Period Sampled	Duration Sampled	Cook Type
Cook 1	HDS Personal Monitor	4001100	Full Cook	2.5 hours	Ether
	DSP Tenax	811-0000038	Full Cook	2.5 hours	Ether
	DSP Carbo Pack X	838-0000211	Full Cook	2.5 hours	Ether
	HDS Personal Monitor	4001101	Full Cook	2.5 hours	Ether
	DSP Tenax	811-0000037	Full Cook	2.5 hours	Ether
	DSP Carbo Pack X	838-0000214	Full Cook	2.5 hours	Ether
	Grab Sample 1	3611	Before Cook	1 minutes	Ether
	Grab Sample 2	3622	During Cook	28 seconds	Ether
	Grab Sample 3	3621	After Cook	30 seconds	Ether
	40mL Vial	1	During Cook	Salt to filter*	Ether
Cook 2	HDS Personal Monitor	4001103	Full Cook	2 hours	Camp Fuel
	DSP Tenax	811-0000039	Full Cook	2 hours	Camp Fuel
	DSP Carbo Pack X	8380000210	Full Cook	2 hours	Camp Fuel
	HDS Personal Monitor	4001098	Full Cook	2 hours	Camp Fuel
	DSP Tenax	811-0000042	Full Cook	2 hours	Camp Fuel
	DSP Carbo Pack X	838-0000215	Full Cook	2 hours	Camp Fuel
	Grab Sample 1	3612	Before Cook	30 seconds	Camp Fuel
	Grab Sample 2	3623	During Cook	30 seconds	Camp Fuel
	Grab Sample 3	3624	After Cook	30 seconds	Camp Fuel
	40mL Vial	2	During Cook	Salt to filter*	Camp Fuel
Cook 3	HDS Personal Monitor	4001106	Full Cook	30 minutes	Camp Fuel
	DSP Tenax	811000041	Full Cook	30 minutes	Camp Fuel
	DSP Carbo Pack X	8380000216	Full Cook	30 minutes	Camp Fuel
	HDS Personal Monitor	4001105	Full Cook	30 minutes	Camp Fuel
	DSP Tenax	8110000036	Full Cook	30 minutes	Camp Fuel
	DSP Carbo Pack X	8380000209	Full Cook	30 minutes	Camp Fuel
	Grab Sample 1	3614	Before Cook	30 seconds	Camp Fuel
	Grab Sample 2	3616	During Cook	30 seconds	Camp Fuel
	Grab Sample 3	3620	After Cook	30 seconds	Camp Fuel
	40mL Vial	3	During Cook	Salt to filter*	Camp Fuel
Cook 4	HDS Personal Monitor	4001107	Full Cook	2 hours	Ether
	DSP Tenax	8110000035	Full Cook	2 hours	Ether
	DSP Carbo Pack X	8380000213	Full Cook	2 hours	Ether
	HDS Personal Monitor	4001099	Full Cook	2 hours	Ether
	DSP Tenax	8110000040	Full Cook	2 hours	Ether
	DSP Carbo Pack X	8380000212	Full Cook	2 hours	Ether
	Grab Sample 1	3618	Before Cook	30 seconds	Ether
	Grab Sample 2	3615	During Cook	30 seconds	Ether
	Grab Sample 3	3621	After Cook	30 seconds	Ether
	40mL Vial	4	During Cook	Salt to filter*	Ether

*The container was opened when salting out the methamphetamine free base from the solvent with the HCl acid gas generator. It was closed when the solid methamphetamine hydrochloride was filtered.

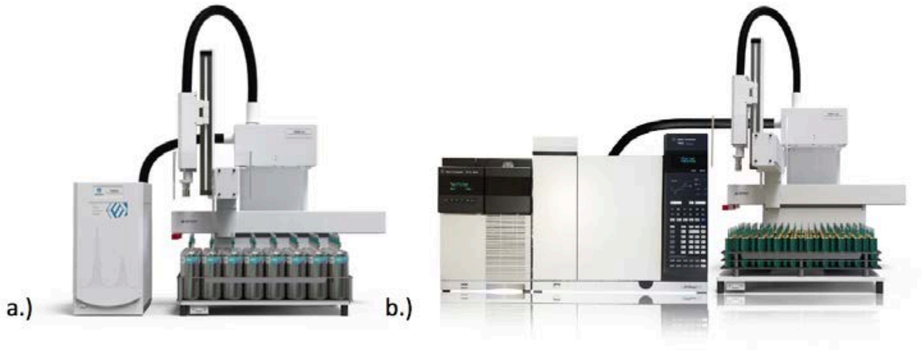


Figure 14. Instrumental set up for analysis of the 1 L MiniCans and HDS personal monitors. a.) The Entech 7200 preconcentratior with the 1 L MiniCans loaded. b.) The Entech 7200 preconcentrator with the HDS personal monitors loaded and a coupled Agilent GC-MS.

Chromatographic separation was achieved with a DB-1 column (60 m x 0.320 mm x 1 µm) by Agilent. The GC was operated in splitless mode. Injection volumes were 1 µL for the 1 L MiniCans^{TM} and 10 µL for the HDS personal monitors. The column oven was programmed as follows: start at 35^{o}C and hold for 5 minutes, increase to 95^{o}C at 6^{o}C/min, increase to 140^{o}C at 10^{o}C/min, and finally increase to 220^{o}C at 15^{o}C/min for a total run time of 24.83 minutes. The MS was set to scan from 29-280 amu at approximately 3 scans per second.

For the DSPs, thermal desorption was used to strip the analytes from the Carbo Pack X and Tenax sorbents. Prior to instrumental analysis, the Carbo Pack X DSPs were spiked with an internal standard mix using an Entech 4200 Sorbent Pen Spiking System. The Tenax DSPs were not spiked with an internal standard mix so all the compounds collected and analyzed from these DSPs were qualitative only. The DSPs were then loaded on to the Entech 5800 Sorbent Pen Desorption Unit that was coupled to an Agilent 7890B GC and an Agilent 5977A MS. This instrumentation is shown in Figure 15.

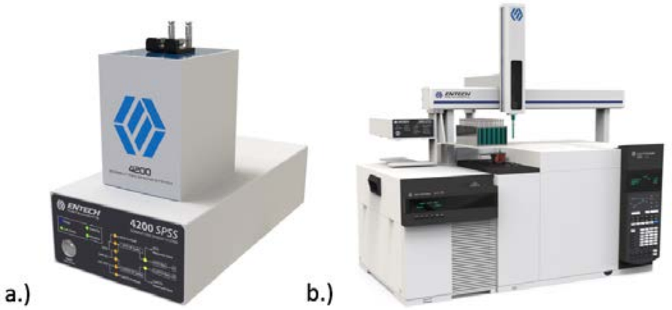


Figure 15. Instrumental set up for analysis of the DSPs, including a.) the Entech 4200 Sorbent Pen Spiking System and b.) the Entech 5800 Sorbent Pen Desorption Unit coupled to an Agilent GC-MS.

Desorption conditions and chromatographic separation were different for the Carbo Pack X and the Tenax sorbents. For the Carbo Pack X sorbent, desorption was achieved by first preheating the DSP at 350°C for 2 minutes, and then desorbing the DSP at 300°C for 5 minutes. Chromatographic separation was achieved with a DB1 pre-column (5 m x 0.530 mm x 0.5 µm) by Agilent followed by a DB-1 column (60 m x 0.320 mm x 1 µm) by Agilent. The GC was operated in split mode with a 25:1 split ratio. The column oven was programmed as follows: start at 35°C and hold for 5 minutes, increase to 150°C at 10°C/min, then increase to 210°C at 15°C/min and hold for 9.5 minutes, for a total run time of 30 minutes. The MS was set to scan from 34-450 amu at 3 scans per second. For the Tenax sorbent, desorption was achieved by first preheating the DSP at 260°C for 2 minutes and then desorbing the DSP at 260°C for 5 minutes. Chromatographic separation was achieved with a silonite coated 0.6m filmless tubing pre-column followed by a DB-5ms Ultra Inert column (30 m x 0.25 mm x 0.5 µm) by Agilent. The GC was operated in split mode with a 10:1 split ratio. The column oven was programmed as follows: start at 35°C and hold for 5 minutes, then increase to 300°C at 10°C/min and hold for 6.5 minutes for a total run time of 38 minutes. The MS was set to scan from 34-450 amu at 3 scans per second.

To analyze the air captured by the 40 mL screw-top vial, internal standards were spiked into the sample through the cap liner. Once internal standards had been added, a headspace sorbent pen (HSP) was inserted into the cap and created a seal with the cap liner. The samples were then evacuated to less than 0.01 atm through the HSP via a Vial Evacuation Tool and placed into a 5600 Sorbent Pen Extraction System (SPES) by Entech. In the SPES, the air samples were subjected to Vacuum Assisted Sorbent Extraction (VASE), which comprised of extracting the air sample into the HSP by placing the sample under vacuum for 20 hours at 35°C. After VASE, the HSP was removed from the vial and analyzed by an Entech 5800 Sorbent Pen Desorption Unit coupled to an Agilent Agilent 7890B GC and an Agilent 5977A MS.

Desorption of the resulting HSP was achieved by first preheating the HSP at 260°C for 2 minutes and then desorbing the HSP at 260°C for 5 minutes. Chromatographic separation was achieved with a silonite coated 0.6m filmless tubing pre-column followed by a DB-5ms Ultra Inert column (30 m x 0.25 mm x 0.5 µm) by Agilent. The GC was operated in split mode with a 10:1 split ratio. The column oven was programmed as follows: start at 35°C and hold for 5 minutes, then increase to 300°C at 10°C/min and hold for 6.5 minutes for a total run time of 38 minutes. The MS was set to scan from 34-450 amu at 3 scans per second.

Standoff Air Monitoring

Standoff air monitoring was performed during each cook to determine the level of ammonia gas, HCl gas, and meth-lab specific air contaminates that could be observed during One Pot methamphetamine production. Instrumentation used for standoff air monitoring included the Geospatial Measurement of Air Pollution (GMAP) unit, sensor pod (SPod) monitors, and a forward looking infrared (FLIR) camera, all provided by the United States Environmental Protection Agency's National Enforcement Investigations Center (EPA-NEIC, Denver, CO), as well as an Ultra-Portable Ammonia and HCl Gas analyzer purchased through Los Gatos Research (LGR-ABB, San Jose, CA).

The GMAP unit, shown in Figure 16, operated by EPA-NEIC during the cooks, is a mobile vehicle equipped with instrumentation meant to aid in industrial gas leak detection and repair. On-board instrumentation includes: an integrative cavity output spectrometer (ICOS) for analysis of methane (CH_4) and carbon dioxide (CO_2); a differential ultraviolet absorption spectrometer (DUVAS) for analysis of benzene, toluene, ethylbenzene, and xylenes (collectively called BTEX); a photo ionization detector (PID) for analysis of volatile organic compounds (VOCs); a global positioning system (GPS) connected to Google Earth Pro (GEP) for sample mapping; a compact meteorological station to monitor wind speed and direction; and an air canister collection mechanism. When the GMAP unit is moving, collected samples can be mapped on GEP to aid in determining the source of monitored air effluents. When the GMAP unit is stationary, collected samples can be used to develop polar plots to map areas of differing air effluent concentrations in order to locate the source of the effluents. If conditions are ideal, the stationary GMAP unit can also be used to estimate the rate of air effluent emissions from a source.

Figure 16. The GMAP unit operated by EPA-NEIC. On-board instrumentation includes an ICOS, PID, DUVAS, GPS, a weather station, and an air canister collection mechanism.

The DUVAS and PID installed on the GMAP unit were used for the detection of ammonia and VOCs respectively. These analytes were monitored during all One Pot methamphetamine cooks and the fire control study, resulting in 8 total data sets. The DUVAS installed on the GMAP unit had been calibrated to measure ammonia when it was assembled in the factory, though no field calibration was performed due to lack of a calibration standard. The PID was calibrated with isobutylene and all results for VOCs were reported as parts per billion (ppb) isobutylene. Table 3 and Figure 17 summarize the locations where samples were collected by the DUVAS and PID installed in the GMAP unit, as well as locations of the SPods that are mentioned in the next section. As can be seen, the GMAP unit was parked approximately 15 m from the One Pot cook shed, and 30 m from the site of the fire control study.

Table 3. GMAP unit and SPod sampling events and locations.

Data Set	Cook	GMAP Location	SPod 25 m Site	SPod 50 m Site	SPod 75 m Site	SPod 100 m Site
1	Ether #1	A	25a	50a	75a	100a
2	Camp Fuel #1	B	25b	50b	75b	100b
3	Camp Fuel Fail #1	C	25b	50b	75b	100b
4	Camp Fuel #2	D	25b	50b	75b	100b
5	Ether #2	C	25b	50b	75b	100b
6	Camp Fuel Fail #2	E	25c	50c	75c	100c
7	Ether #3	E	25c	50c	75c	100c
8	Fire Control Study	F	25c	50c	75c	100c

Figure 17. GMAP unit and SPod sampling locations at the Oklahoma State University Fire Research and Training Center.

The SPods provided by EPA-NEIC, shown in Figure 18, have been developed as a stationary, complementary instrument for gas leak detection and repair. The SPods contain a PID set to measure VOCs, as well as a meteorological station to monitor wind speed and direction. The solar-powered, stationary SPods can develop polar plots in the same manner as the GMAP unit while it is stationary. For this study, 4 SPods were deployed downwind of the One Pot methamphetamine cooks at graduated distances from the cook shed. Three of the SPods were developed by the EPA's Office of Research and Development (EPA-ORD, Research Triangle Park, NC) and the fourth SPod is a commercially available SPod developed by SenSevere Environmental Sensors (SenSevere LLC, Pittsburgh, PA). The approximate radial standoff distances that the SPods were placed in-line from the cook shed were 25, 50, 75, and 100 meters (m). As with the GMAP unit, the SPods were deployed during all 6 successful One Pot methamphetamine cooks, the 2 One Pot bottle-failures, and the fire control study, resulting in 8 total data sets. For the first two cook days, SPod A was deployed at 25 m, SPod B was deployed at 50 m, and SPod B was deployed at 75 m. For the final cook day, SPod C was deployed at 25 m, SPod A was deployed at 50 m, and SPod B was deployed at 75 m. The SenSevere SPod was always deployed at 100 m. Table 3 and Figure 17 summarize the locations were the SPods were deployed during the One Pot methamphetamine cooks.

Figure 18. Three SPods deployed at 50, 75, and 100 m from the One Pot methamphetamine cook shed.

A GF320 infrared imaging video camera designed by FLIR Systems (FLIR Systems Inc, Wilsonville, OR), shown in Figure 19, was used to visualize the volatile effluents being emitted by the One Pot methamphetamine cooks. VOCs could be visualized with the FLIR camera due to a bandpass filter implemented into the camera that limited the image to a narrow band of the IR light spectrum. To enhance the visualization of the VOCs, the FLIR camera was set to high sensitivity mode. In high sensitivity mode, part of the image is subtracted in order to better highlight movements, such as VOCs being emitted from the One Pot methamphetamine cook.

Figure 19. GF320 FLIR camera designed by FLIR Systems Inc.

A portable Integrated Cavity Output Spectroscopy (ICOS) gas analyzer (Los Gatos Research, San Jose, CA) was also used to detect ammonia plumes generated by the One Pot methamphetamine labs. The ICOS was designed to detect ammonia gas in the range of 10 ppb - 200 ppm simultaneously with hydrogen chloride gas in the range of 0.3 to 2,000 ppb and also water vapor. The ICOS internal optical cavity utilizes continuous-wave lasers to data-log ppb-level NH_3 and HCl using an external diaphragm vacuum pump and was used both plug-connected when stationary and also powered via battery packs, especially during vehicle passes in driving circuit loops made tangential to the cook shed, as shown in Figure 20. The special-built, combined NH_3-HCl unit was factory calibrated and demonstrated good response to ammonia, but HCl detection was highly suppressed and recorded data is not used in this report. During each cook, wind direction was carefully observed and ICOS placement was pre-planned to be in the most advantageous downwind position. Slight wind shifts were observed by the nearby windsock during data collection, especially during the Wednesday cooks (See Figure 21).

Figure 20. Configuration of battery-powered ICOS NH_3-HCl gas analyzer (foreground), vacuum pump (gray), battery pack (yellow-right) as positioned in moving vehicle during data-logging. Trace ammonia detected from vehicle exhaust was dampened or eliminated by opening all vehicle windows when vehicle was in motion or by shutting off the engine when parked as shown.

Figure 21. Wind sock seen above 100 m ICOS placement, with SPod visible left and cook shed out of view to left of frame.

Fire Control Study

To assess the effectiveness of several popular firefighting techniques used by law enforcement to control One Pot methamphetamine labs fires, the cook waste bottles with the contained solvent and waste heel from the One Pots were set on fire at the conclusion of the study, and the resulting fire was treated with Cold Fire^{TM} Tactical (Cold Fire Tactical LTD, Lenexa, KS), Imbiber Beads® (Imbibitive Technologies America Inc, Newark DE), and a dry-powder training fire extinguisher loaded with sodium bicarbonate. Each technique was tested on a burning One Pot that used ether as the organic solvent and a One Pot that used camp fuel as the organic solvent. The One Pots were ignited by a trained firefighter who was wearing turnout gear and SCBA. As shown in Figure 22, all One Pots were ignited with a propane torch. Results from this fire control study were subjective only, with personnel observing these controlled burns reporting their individual visual conclusions on how well each firefighting technique contained and/or doused each fire, with no scientific instruments deployed or variables measured to rank each technique.

Figure 22. All One Pots were ignited by a trained firefighter wearing turnout gear and SCBA. One Pots were ignited with a propane torch as shown.

Results and Discussion

One Pot Cooks

Seven One Pot methamphetamine cooks were performed from Tuesday, November 28, 2017 through Thursday, November 30, 2017, six planned, plus one additional cook following a bottle failure during a camp fuel cook. The cook performed the morning of Wednesday, November 29, experienced a bottle failure during the repeated over-pressurization and depressurization of the plastic bottle (burping phase). This repeated expansion and relaxation of the plastic led to the formation of a small hole in the bottle (See Figure 23). Under the pressure created within the reaction vessel of the One Pot, organic solvent was rapidly sprayed out of the hole, resulting in the loss of methamphetamine from the cook and presenting a potential fire hazard due to exposure of the lithium strips to air. Following this event, a new camp fuel cook was commenced following cleanup of the bottle failure and resulting spillage.

Figure 23. A hole formed in the One Pot reaction vessel of the second camp fuel cook due to repeated expansion and relaxation of the plastic.

As shown in Figure 24, a second bottle failure occurred on the morning of Thursday, November 30, during the third camp fuel cook. Like the first bottle failure, repeated expansion and relaxation of the plastic bottle led to a small hole opening in the One Pot reaction vessel. It is believed that both bottle failures occurred following addition of sodium hydroxide when a lack of "rolling" (visible bubbling and agitation) was observed during the cook. The additional sodium hydroxide likely led to a greater formation of ammonia gas and thus greater pressures within the plastic bottles, ultimately leading to the bottle failures. Due to the limited amount of resources available at the research cook site, another camp fuel One Pot could not be performed after the second bottle failure. Instead, as can be seen in Figure 25, what could be salvaged from the second bottle failure was added to a 64 oz. rectangular, deep dish Glad food storage container (Glad Products Company, Oakland, CA) along with the entire contents of a new instant cold compress pack and a capful of Drain Out, and the cook was allowed to proceed as if no bottle failure had occurred. For burping, the lid was taken off the Glad container and the solid found at the bottom of the One Pot was stirred with a plastic spatula. All other procedures mirrored that of the other One Pot methamphetamine cooks.

Figure 24. A hole formed in the One Pot reaction vessel of the final camp fuel cook due to repeated expansion and relaxation of the plastic.

Figure 25. A One Pot methamphetamine lab performed in a 64 oz. Glad food storage container after the original bottle failed during the final camp fuel cook.

Surface Contamination Sampling

A total of 96 lateral flow immunoassays were performed on swab samples collected from various surfaces within the cook shed, directly outside the cook shed, and on the protective suits worn by the researchers during the One Pot methamphetamine cooks, as well as on the clothing worn by two researchers who collected the swab samples throughout this project. The swab samples were taken at various times throughout the One Pot methamphetamine cooks, including before the cook (pre-cook), after the cook (post-cook), and after a wet decontamination procedure was performed (post-decon). The results of the LFIAs performed on the surface swab samples are provided in Table 4. As described above, if two lines were observed on the LFIA, the swab sample was deemed negative for methamphetamine at a limit of detection (LOD) of 50 ng/100 cm^2. If only one line was observed on the LFIA, the swab sample was deemed positive for methamphetamine. If one line was clearly observed and the second was faintly observed, the sample was deemed positive at a level near that of the LOD and was designated as a trace positive. Following cook 1, only 2 additional pre-cook surface swab samples were collected. This was due to the assumption that if the surfaces were positive post-cook, they would remain positive prior to the next cook, and if surfaces were negative prior to the wet decontamination procedure, the surfaces would remain negative at the start of the next One Pot methamphetamine cook.

Table 4. Results of the lateral flow immunoassays and fluorescence covalent microbead immunosorbent assays performed on the surface swab samples from within the One Pot methamphetamine cook shed, directly outside the cook shed, and on the protective suits worn by the researchers during the One Pot methamphetamine cooks. As described above, LFIA and FCMIA results were derived from divided liquid fractions taken from the same sample solution.

	Sample ID	Sample Location	Sample Time	LFIA Results	FCMIA Results (ng/100 cm^2)	Cook Type
Cook 1	LFIA 1A	Left Side Shelf	Pre-Cook	Negative	0.15	Ether
	LFIA 1B	Left Side Shelf	Post-Cook	Positive	6.20	Ether
	LFIA 2A	Right Side Shelf	Pre-Cook	Negative	0.14	Ether
	LFIA 2B	Right Side Shelf	Post-Cook	Positive	7.94	Ether
	LFIA 3A	Left Front Table	Pre-Cook	Negative	0.26	Ether
	LFIA 3B	Left Front Table	Post-Cook	Negative	257.69	Ether
	LFIA 4A	Right Front Table	Pre-Cook	Negative	1.36	Ether
	LFIA 4B	Right Front Table	Post-Cook	Positive	130.82	Ether
	LFIA 5A	Left Wall	Pre-Cook	Negative	0.60	Ether
	LFIA 5B	Left Wall	Post-Cook	Trace	3.92	Ether
	LFIA 6A	Back Table	Pre-Cook	Negative	0.83	Ether
	LFIA 6B	Back Table	Post-Cook	Positive	9.04	Ether
	LFIA 7A	Back Wall	Pre-Cook	Negative	0.50	Ether
	LFIA 7B	Back Wall	Post-Cook	Positive	1.35	Ether
	LFIA 8A	Right Wall	Pre-Cook	Negative	0.31	Ether

	LFIA 8B	Right Wall	Post-Cook	Positive	2.05	Ether
	LFIA 9A	Floor Back	Pre-Cook	Negative	0.57	Ether
	LFIA 9B	Floor Back	Post-Cook	Positive	1.00	Ether
	LFIA 10A	Ceiling	Pre-Cook	Negative	0.29	Ether
	LFIA 10B	Ceiling	Post-Cook	Positive	1.98	Ether
	LFIA 11A	Floor Front	Pre-Cook	Negative	0.53	Ether
	LFIA 11B	Floor Front	Post-Cook	Positive	4.34	Ether
	LFIA 12A	Left Door	Pre-Cook	Negative	0.39	Ether
	LFIA 12B	Left Door	Post-Cook	Positive	2.57	Ether
	LFIA 13A	Right Door	Pre-Cook	Negative	2.41	Ether
	LFIA 13B	Right Door	Post-Cook	Negative	0.10	Ether
	LFIA 14A	Front Right Arm-Researcher 2	Pre-Cook	Negative	0.12	Ether
	LFIA 14B	Front Right Arm-Researcher 2	Post-Cook	Negative	1.59	Ether
	LFIA 15A	Front Left Arm-Researcher 2	Pre-Cook	Negative	0.26	Ether
	LFIA 15B	Front Left Arm-Researcher 2	Post-Cook	Negative	0.83	Ether
	LFIA 16A	Back Right Leg-Researcher 2	Pre-Cook	Negative	0.21	Ether
	LFIA 16B	Back Right Leg-Researcher 2	Post-Cook	Negative	0.76	Ether
	LFIA 17A	Back Left Leg-Researcher 2	Pre-Cook	Negative	0.21	Ether
	LFIA 17B	Back Left Leg-Researcher 2	Post-Cook	Positive	2.48	Ether
	LFIA 18A	Back Head-Researcher 2	Pre-Cook	Negative	0.16	Ether
	LFIA 18B	Back Head-Researcher 2	Post-Cook	Negative	0.68	Ether
	LFIA 19A	Front Right Arm-Researcher 1	Pre-Cook	Negative	0.15	Ether
	LFIA 19B	Front Right Arm-Researcher 1	Post-Cook	Positive	6.52	Ether
	LFIA 20A	Front Left Arm-Researcher 1	Pre-Cook	Negative	0.19	Ether
	LFIA 20B	Front Left Arm-Researcher 1	Post-Cook	Positive	2.41	Ether
	LFIA 21A	Back Right Leg-Researcher 1	Pre-Cook	Negative	0.15	Ether
	LFIA 21B	Back Right Leg-Researcher 1	Post-Cook	Positive	16.32	Ether
	LFIA 22A	Back Left Leg-Researcher 1	Pre-Cook	Negative	0.13	Ether
	LFIA 22B	Back Left Leg-Researcher 1	Post-Cook	Positive	3.91	Ether
	LFIA 23A	Face Mask-Researcher 1	Pre-Cook	Negative	0.14	Ether
	LFIA 23B	Face Mask-Researcher 1	Post-Cook	Positive	7.30	Ether
	LFIA 24A	Field Blank	-	Negative	0.13	Ether
	LFIA 25B	Field Blank	-	Negative	0.12	Ether
Cook 2	LFIA 29A	Front Right Arm-Researcher 3	Post-Cook	Negative	1.18	Camp Fuel
	LFIA 30A	Front Left Arm-Researcher 3	Post-Cook	Negative	0.67	Camp Fuel
	LFIA 31A	Back Left Leg-Researcher 3	Post-Cook	Negative	0.41	Camp Fuel
	LFIA 32A	Back Right Leg-Researcher 3	Post-Cook	Negative	0.64	Camp Fuel
	LFIA 33A	Forehead-Researcher 3	Post-Cook	Positive	5.26	Camp Fuel
	LFIA 34A	Front Right Arm-Researcher 4	Post-Cook	Positive	4.10	Camp Fuel
	LFIA 35A	Front Left Arm-Researcher 4	Post-Cook	Trace	4.90	Camp Fuel
	LFIA 36A	Back Left Leg-Researcher 4	Post-Cook	Negative	1.76	Camp Fuel
	LFIA 37A	Back Right Leg-Researcher 4	Post-Cook	Negative	3.55	Camp Fuel
	LFIA 38A	Face Mask-Researcher 4	Post-Cook	Positive	9.84	Camp Fuel

Cook 3	LFIA 29B	Air Tank-Researcher 1	Post-Decon	Negative	0.45	Camp Fuel
	LFIA 30B	Left Side Shelf	Post-Decon	Negative	1.13	Camp Fuel
	LFIA 31B	Right Side Shelf	Post-Decon	Negative	0.62	Camp Fuel
	LFIA 32B	Left Front Table	Post-Decon	Trace	4.30	Camp Fuel
	LFIA 33B	Right Front Table	Post-Decon	Positive	45.67	Camp Fuel
	LFIA 34B	Left Wall	Post-Decon	Negative	1.32	Camp Fuel
	LFIA 35B	Back Table	Post-Decon	Negative	3.17	Camp Fuel
	LFIA 36B	Back Wall	Post-Decon	Negative	0.65	Camp Fuel
	LFIA 37B	Right Wall	Post-Decon	Negative	1.04	Camp Fuel
	LFIA 38B	Floor Back	Post-Decon	Negative	1.31	Camp Fuel
	LFIA 39B	Ceiling	Post-Decon	Negative	1.71	Camp Fuel
	LFIA 40B	Floor Front	Post-Decon	Negative	2.47	Camp Fuel
	LFIA 41B	Left Door	Post-Decon	Negative	1.29	Camp Fuel
	LFIA 42B	Right Door	Post-Decon	Negative	1.10	Camp Fuel
	LFIA 39A	Front Right Leg-Researcher 2	Post-Cook	Positive	52.38	Camp Fuel
	LFIA 40A	Front Right Leg-Researcher 2	Post-Decon	Negative	1.67	Camp Fuel
Cook 4	LFIA 41A	Air Tank-Researcher 1	Post-Decon	Negative	1.49	Ether
	LFIA 42A	Air Tank-Researcher 4	Post-Decon	Negative	1.99	Ether
	LFIA 43A	Chest-Researcher 3	Post-Cook	Positive	15.45	Ether
	LFIA 44A	Right Front Table	Post-Cook	Positive	264.35	Ether
	LFIA 43B	Chest-Researcher 3	Post-Decon	Negative	1.05	Ether
	LFIA 45A	Left wall	Post-Cook	Trace	8.60	Ether
	LFIA 46A	Left door	Post-Cook	Trace	9.86	Ether
Cook 5	LFIA 44B	Right Front Table	Post-Decon	Positive	37.20	Camp Fuel
	LFIA 45B	Left Wall (clean)	Post-Decon	Negative	1.53	Camp Fuel
	LFIA 46B	Left Door	Post-Decon	Negative	1.70	Camp Fuel
	LFIA 47A	Air Tank-Researcher 1	Post-Decon	Negative	0.52	Camp Fuel
	LFIA 48A	Air Tank-Researcher 4	Post-Decon	Negative	0.27	Camp Fuel
	LFIA 48B	Air tank-Researcher 4	Post-Cook	Trace	5.53	Camp Fuel
	LFIA 49A	Chest-Researcher 3	Post-Decon	Trace	22.17	Camp Fuel
	LFIA 49B	Chest-Researcher 3	Post-Cook	Negative	1.62	Camp Fuel
Cook 6	LFIA 50A	Belt on Air Tank-Researcher 1	Post-Cook	Positive	21.26	Ether
	LFIA 50B	Belt on Air Tank-Researcher 1	Post-Decon	Negative	2.50	Ether
	LFIA 51A	OBNDD Suit	-	Negative	1.62	Ether
	LFIA 51B	Right Front Table	Post-Decon	Trace	15.43	Ether
	LFIA 52A	Left Wall	Post-Decon	Negative	0.85	Ether
	LFIA 28A	Jeans-Sample Collector 1	-	Negative	0.35	Ether
	LFIA 28B	Jeans-Sample Collector 2	-	Negative	0.39	Ether

Table 5 summarizes the results of the LFIAs performed on the surface swab samples. Of the 96 LFIAs performed, 25 were positive for methamphetamine, 8 had trace levels of methamphetamine observed, and 63 were negative for methamphetamine. Table 6 shows this data broken up by the time period in which the surface swab sample was collected. When the data is broken up by time periods, it is observed that all 23 samples collected prior to production of a One Pot methamphetamine lab were negative for methamphetamine. For the 41 surface swab samples collected after the production of the One Pot methamphetamine labs, 23 were positive for methamphetamine, 5 produced a trace result for methamphetamine, and 13 were negative for methamphetamine. For the 27 surface swab samples collected immediately following the wet decontamination procedure, 2 were still positive for methamphetamine, 3 produced a trace result for methamphetamine, and 20 were negative for methamphetamine, suggesting the wet decontamination procedure removed surface methamphetamine contamination with an 80% success rate.

Table 5. Summary of the results from the lateral flow immunoassays performed on the surface swab samples.

	Positive	**Trace**	**Negative**	**Total**
Cook 1	16	1	31	48
Cook 2	3	1	6	10
Cook 3	2	1	13	16
Cook 4	2	2	3	7
Cook 5	1	2	5	8
Cook 6	1	1	5	7
Total	25	8	63	96

Table 6. Lateral flow immunoassay results broken up by the time period that the surface swab sample was collected.

	Positive	**Trace**	**Negative**	**Total**
Pre-Cook	0	0	23	23
Post-Cook	23	5	13	41
Post-Decon	2	3	22	27
Other	0	0	5	5
Total	25	8	63	96

The quantitative results for the surface swab samples is provided in Table 4. As can be seen, many of the samples that were deemed positive by the LFIA had concentrations of methamphetamine below 50 ng/100 cm^2, suggesting the FLIA's LOD of 50 ng/100 cm^2 may be overly conservative. Of the 96 surface swabs analyzed, only one sample presented a false negative via FLIA. Sample LFIA 3B came up negative during the presumptive FLIA test that was performed in the field, but this same sample was found to have a methamphetamine concentration of 257.69 ng/100 cm^2 when analyzed at the CDC-NIOSH laboratory via FCMIA. No samples, including the field blanks, were found to be completely absent of methamphetamine.

A summary of the quantitative results from the surface swab samples is given in Table 7. The lowest level of methamphetamine observed from these samples was 0.10 ng/100 cm^2, which was observed in the post-cook sample FLIA 13B, while the highest level of methamphetamine observed from these samples was 264.35 ng/100 cm^2, which was observed in the post-cook sample FLIA 44A. The average methamphetamine concentrations observed were 0.44, 21.54, and 5.73 ng/100 cm^2 for the pre-cook samples, post-cook samples, and post-decon samples respectively.

Table 7. Summary of the FCMIA quantitative results from the surface swab samples.

	Pre-Cook	**Post-Cook**	**Post-Decon**
Max Conc.	2.41	264.35	45.67
Min Conc.	0.12	0.10	0.27
Mean Conc.	0.44 (±0.52)^b	21.54 (±58.91)^a	5.73 (±11.41)^{ab}

*2 means with the same letter are not significantly different at α=0.05.

Shed Interior Air Monitoring

Active Air Sampling

Of the 27 PTFE filters sent to the CDC-NIOSH laboratory in Cincinnati, OH, 26 were analyzed for the presence of methamphetamine. Filter number 1 was sacrificed to verify the quantitative method, and therefore no data was obtained from this filter. The concentration of methamphetamine captured by each filter is given in Table 8, with the concentration of methamphetamine listed in micrograms of methamphetamine per cubic meter of air. For the first camp fuel cook and the second ether cook, a higher amount of methamphetamine per cubic foot of air was captured by the PTFE filters from the active samplers that were turned off during the cook and only drew in air during the final salting out process. One possible explanation for the higher methamphetamine seen by the filters exposed only during the salting out stage is that the air sample filters that were exposed throughout the entire cook may have become surface-saturated with other volatile One Pot methamphetamine lab byproducts, thus preventing them from fully capturing methamphetamine, which would be more likely to volatilize during the salting out step of the cook process when the One Pot vessel is depressurized and open to atmosphere.

Table 8. The concentration of methamphetamine captured by the PTFE filters loaded into the SKC Airchek 2000 active air samplers.

	Filter Number	Pump Location	Activated Period	Cook Type	Methamphetamine Conc. (μg/m^3)
Cook 2	1	Front Left	Full	Camp Fuel	NA
	2	Back Right	Full	Camp Fuel	0.31
	3	Researcher	Full	Camp Fuel	0.87
	4	Front Left	Salting Out	Camp Fuel	3.51
	5	Back Right	Salting Out	Camp Fuel	2.41
	6	Field Blank	Flash Exposure	Field Blank	0.00
Cook 3	7	Front Left	Full	Camp Fuel	2.01
	8	Back Right	Full	Camp Fuel	1.68
	9	Researcher	Full	Camp Fuel	1.35
	10	Front Left	Salting Out	Camp Fuel	0.52
	11	Back Right	Salting Out	Camp Fuel	0.02
Cook 4	12	Researcher	Full	Ether	3.59
	13	Front Left	Full	Ether	0.69
	14	Back Right	Full	Ether	0.40
	15	Back Right	Salting Out	Ether	12.75
	16	Front Left	Salting Out	Ether	2.49
Cook 5	17	Back Right	Full	Camp Fuel	12.75
	18	Front Left	Full	Camp Fuel	1.75
	19	Researcher	Full	Camp Fuel	3.18
	20	Back Right	Salting Out	Camp Fuel	0.31
	21	Front Left	Salting Out	Camp Fuel	1.25
Cook 6	22	Front Left	Full	Ether	2.43
	23	Back Right	Full	Ether	1.43
	24	Researcher	Full	Ether	0.06
	25	Front Left	Salting Out	Ether	0.00
	26	Back Right	Salting Out	Ether	0.00
	27	Field Blank	Flash Exposure	Field Blank	0.00

Passive Air Sampling

As described above, 1-liter vacuum canister grab samples were collected at 3 points in the One Pot methamphetamine cooks and laboratory analyzed by Entech using GC-MS. Due to a lack of chemical reference standards available for this research, observed chromatographic peaks were identified based on a mass spectra library search using the NIST mass spectra data base instead of the standard practice of comparing an unknown peak to a known reference standard. The 82 compounds included in the EPA's TO-14A, TO-15, and BTEX compound list were quantified using an external calibration curve and all other compounds were only identified, though some can be semi-quantified by estimating their concentrations by comparing the compounds peak area to the peak area from one of the 82 quantified compounds. Methamphetamine and methamphetamine-related byproducts were not identified in the chromatograms of any of the passive air samples in this study.

For the 1 L MiniCans^{TM}, 21 quantifiable compounds were identified from the 12 air samples. A summary of the compounds quantified and their concentration in parts per billion (ppb) air is shown in Table 9. Each MiniCan^{TM} air sample was given a name that follows the following template: Day-Cook-Time, so sample D1-C1-Pre means the sample was collected on the first day, during the first cook, prior to the cook being performed. As described above, the 1 L vacuum canister grab samples were collected at 3 points throughout the One Pot methamphetamine cooks: "Pre" vacuum grabs were taken prior to the start of the cooks, "Mid" indicates after the cook reaction was complete but before salting out began, and "Post" means after the powdered methamphetamine had been separated from the post-salt solvent via filtration. The acetone observed in the MiniCan^{TM} air sample prior to the first ether cook was due to the air sample being collected after the starting fluid cans had been depressurized, resulting in a small amount of VOCs being present in the air. The presence of cyclohexane and heptane in the air samples collected prior to the second ether cook are attributed to the bottle failure that occurred during the morning. The bottle failure caused camp fuel to be sprayed throughout the cook shed, resulting in a continuous source of VOCs throughout the early part of the afternoon when the second ether cook was being performed. The data shows that by mid-cook, the camp fuel had evaporated and the VOCs from that solvent had fallen below the limit of quantitation for this study.

A trend that was observed with the MiniCan^{TM} air data presented in Table 9 is that the VOCs captured during the camp fuel One Pot methamphetamine cooks quickly dissipated and the concentrations decreased approximately tenfold between the mid-cook sample and the post-cook sample. However, during the ether cooks, the concentrations of some VOCs captured by the MiniCans^{TM}, increased slightly between the mid-cook samples and the post-cook samples, while ether decreased. As can be seen in the gas chromatograms in Figures 26-29, this same trend can be observed for several of the semi-quantitated compounds, namely the methylated hydrocarbons with carbon chain lengths of 5-7.

Table 9. Summary of the quantifiable VOCs collected with the 12 MiniCans^{TM}. All concentrations are in ppb air.

Cook	**Ether 1**			**Camp Fuel 1**			**Fail 1/Camp Fuel 2**			**Ether 2**		
Description (Day, Cook, Time)	D1-C1-Pre	D1-C1-Mid	D1-C1-Post	D1-C2-Pre	D1-C2-Mid	D1-C2-Post	D2-C1-Pre	D2-C1-Mid	D2-C1-Post	D2-C2-Pre	D2-C2-Mid	D2-C2-Post
Acetone	0.80	1.09	0.89	3.13	-	3.10	0.98	-	-	-	0.59	0.69
Isopropyl Alcohol	-	3.21	-	-	-	-	-	1.04	-	-	-	-
Ethyl ether	-	83.78	31.28	-	-	-	-	-	-	-	84.18	7.06
Heptane	-	54.30	88.63	-	45.14	4.08	-	213.95	20.70	1.13	10.03	20.19
3-Methyl hexane	-	0.55	0.78	-	-	-	-	1.26	-	-	-	-
Methyl cyclohexane	-	0.75	1.26	-	3.58	-	-	18.72	1.79	-	-	-
Cyclohexane	-	-	1.20	-	133.26	4.57	-	585.36	43.05	1.66	-	-
Propane	-	-	-	-	6.26	0.52	-	2.61	-	-	-	-
2-Methyl pentane	-	-	-	-	0.75	-	-	1.70	-	-	-	-
Pentane	-	-	-	-	16.14	-	-	15.91	4.43	-	-	-
2-Methyl butane	-	-	-	-	823.19	2.61	-	633.03	195.65	1.62	-	-
Isopropylcyclobutane	-	-	-	-	-	-	-	1.11	-	-	-	-
1,3-Dimethylcyclopentane	-	-	-	-	-	-	-	1.31	-	-	-	-
3,3-Dimethyl hexane	-	-	-	-	-	-	-	0.71	-	-	-	-
2-Methyl heptane	-	-	-	-	-	-	-	10.32	1.47	-	-	-
1,2-Dimethyl cyclohexane	-	-	-	-	-	-	-	1.84	-	-	-	-
Propyl cyclohexane	-	-	-	-	-	-	-	0.61	-	-	-	-
Hexane	-	-	-	-	2.67	-	-	7.65	0.73	-	-	-
3-Ethyl-2-pentanol	-	-	-	-	-	-	-	-	0.69	-	-	-

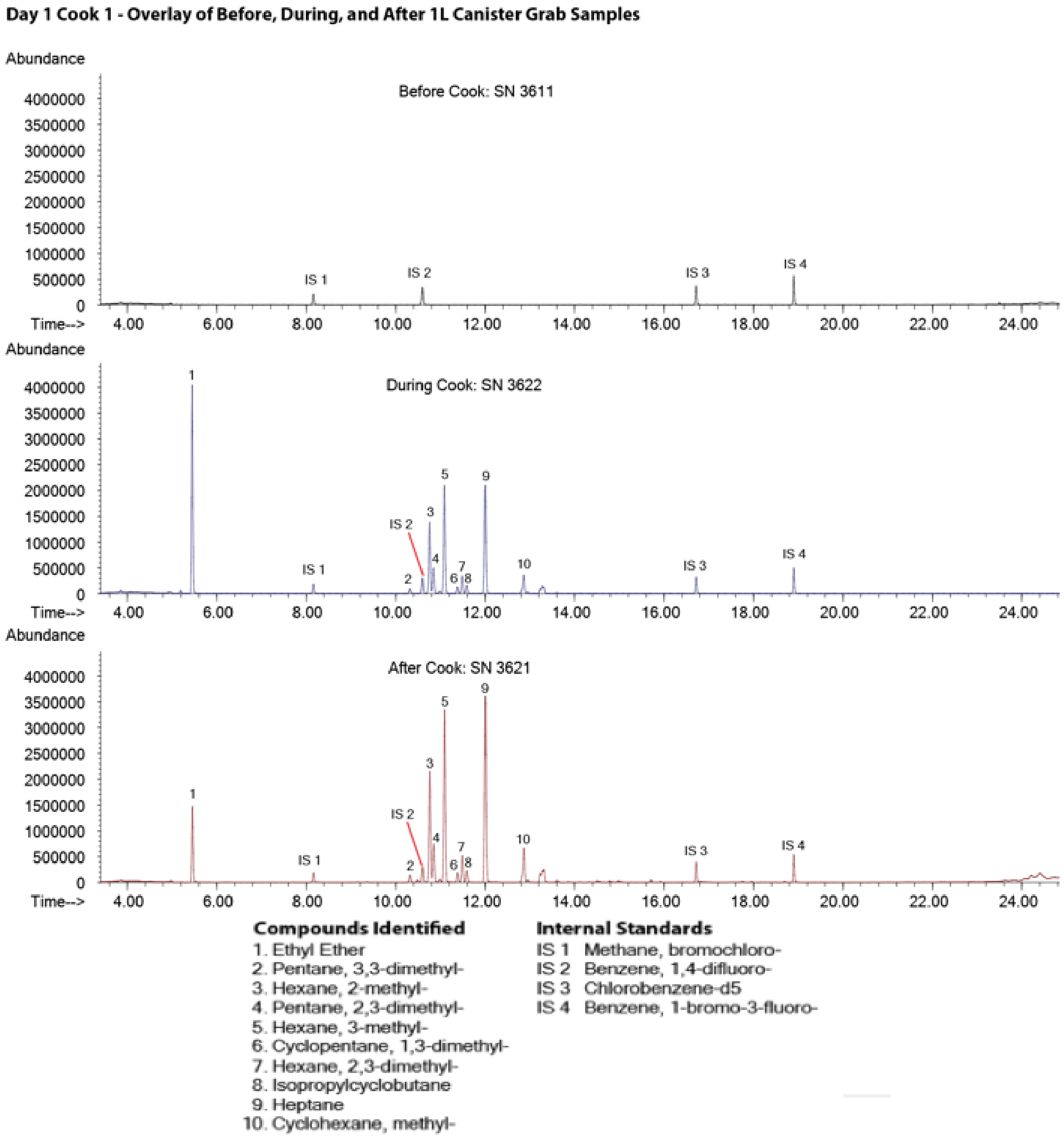


Figure 26. Gas chromatograms from the 3 MiniCan™ air samples taken during the first ether cook.

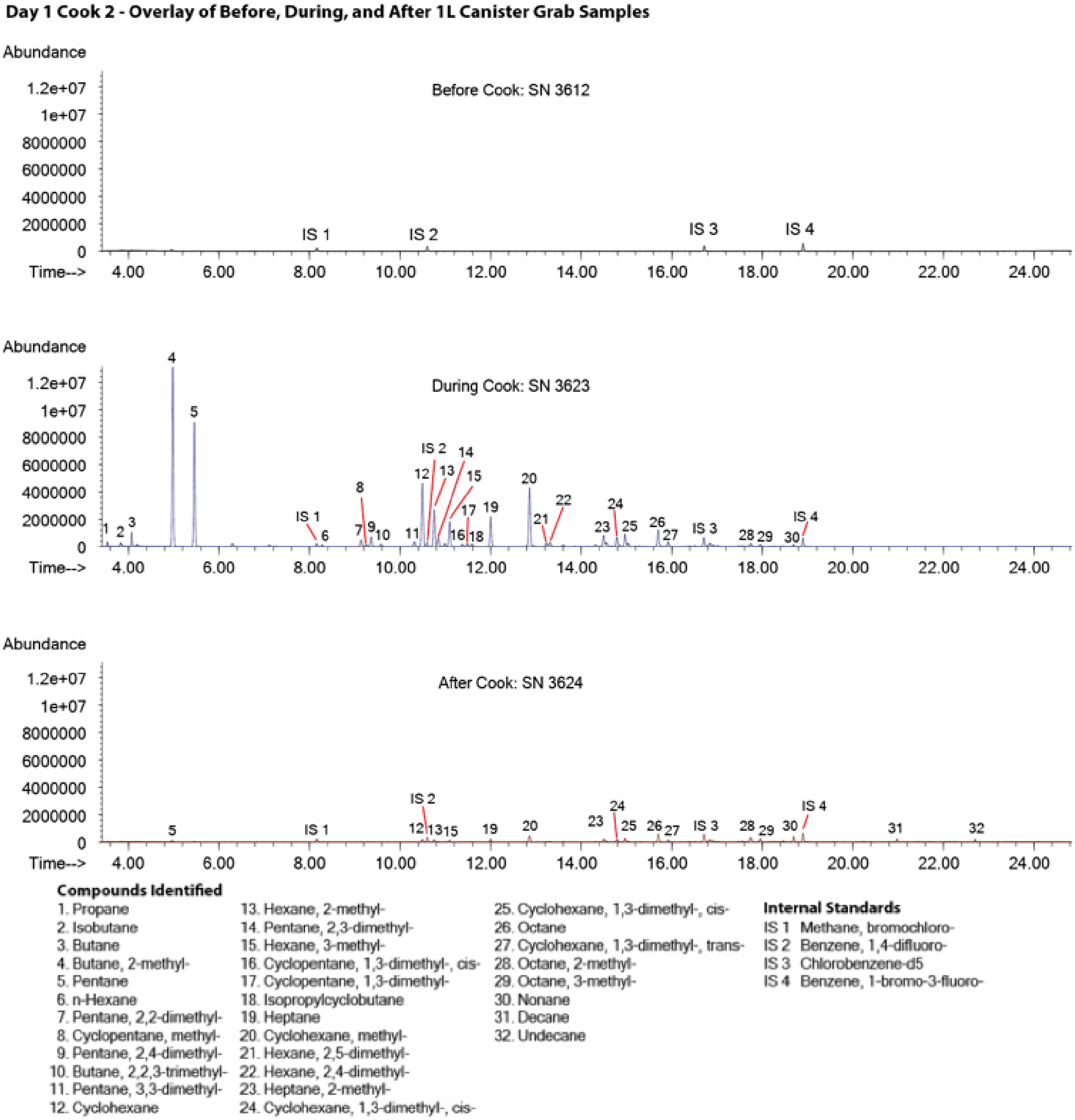


Figure 27. Gas chromatograms from the 3 MiniCan^{TM} air samples taken during the first camp fuel cook.

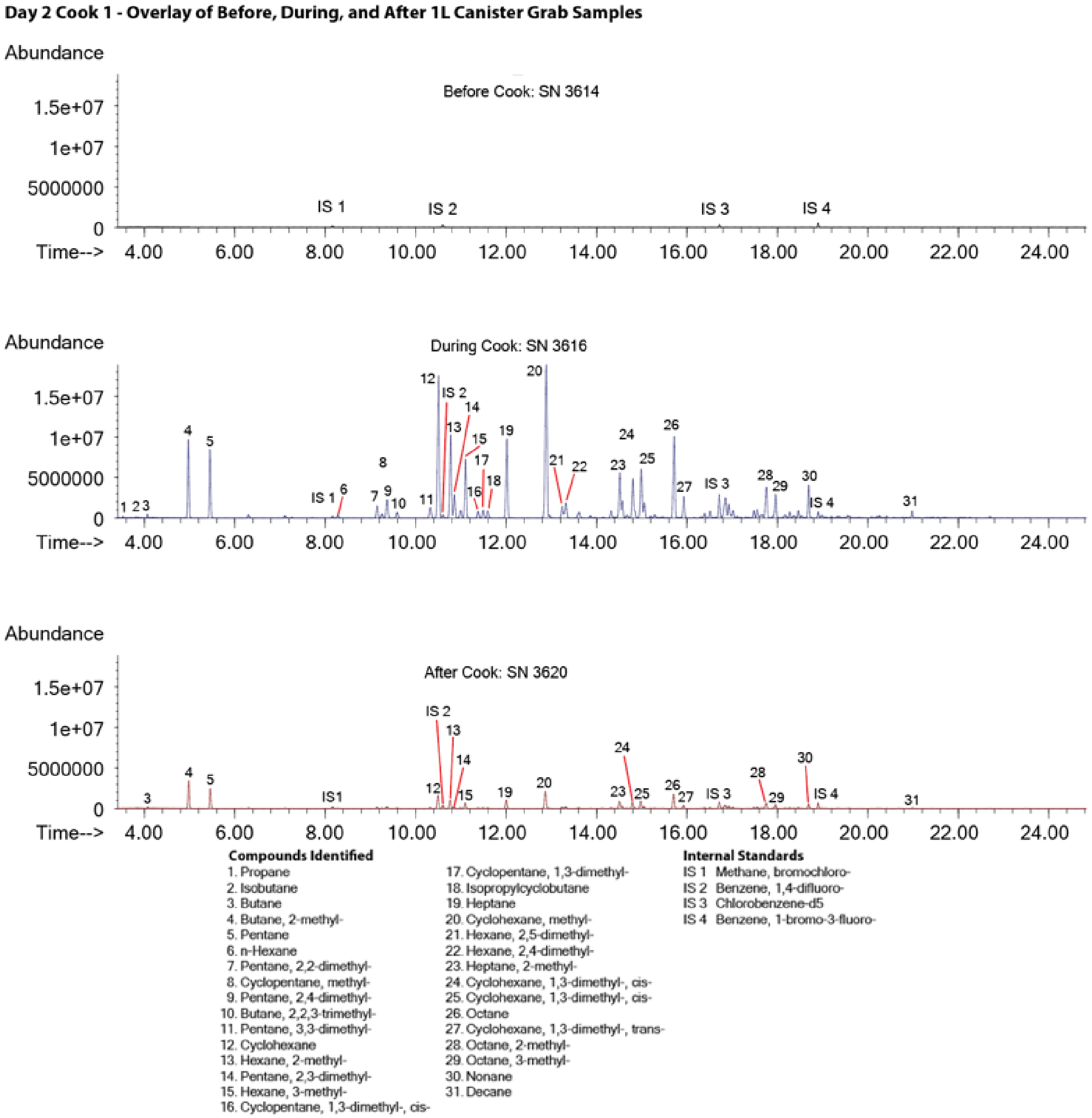


Figure 28. Gas chromatograms from the 3 MiniCan™ air samples: top chromatogram taken pre-cook prior to the first camp fuel bottle failure, middle prior to salting out during the second (successful) camp fuel cook, and bottom following conclusion of second cook.

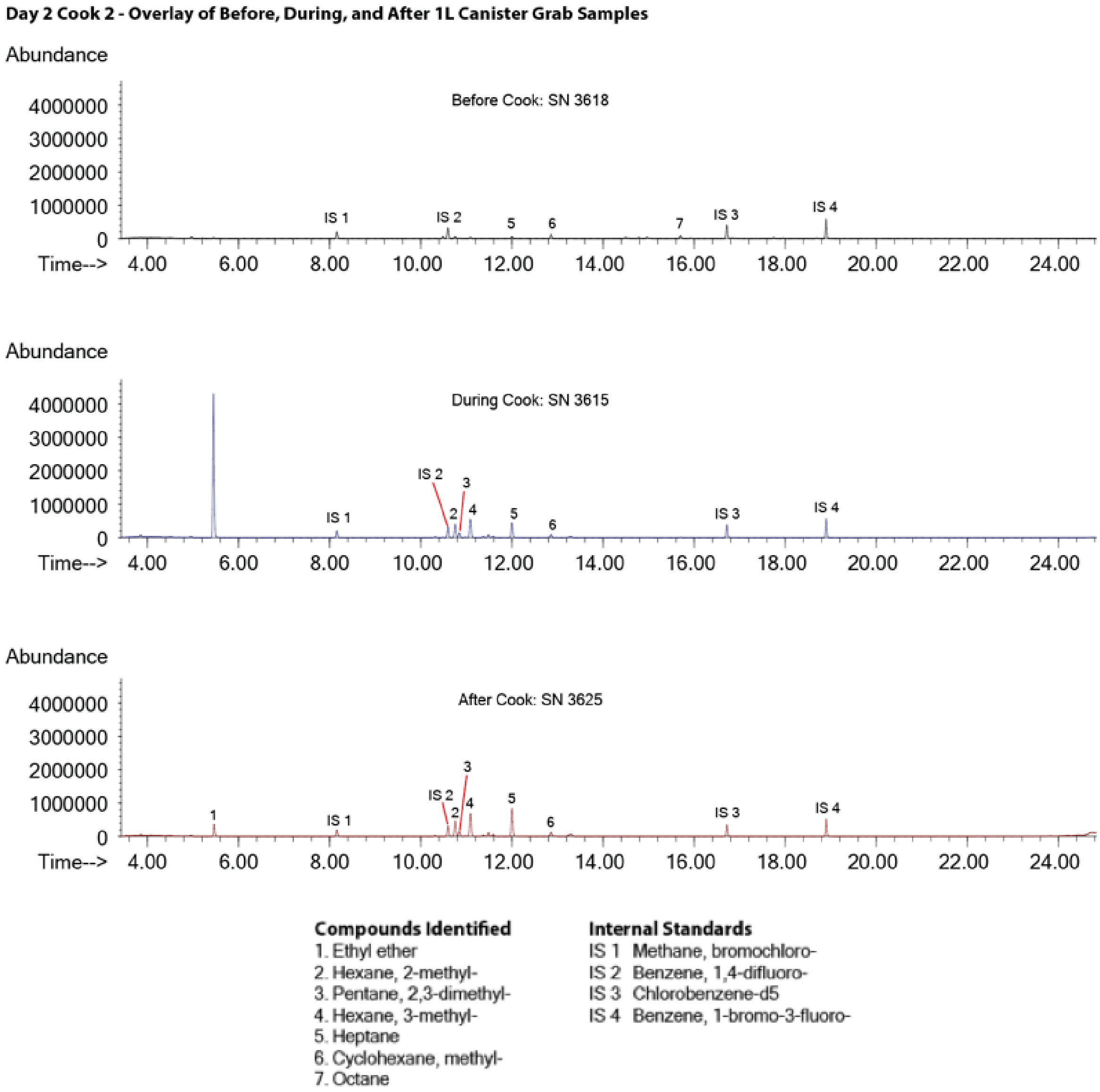


Figure 29. Gas chromatograms from the 3 MiniCan^{TM} air samples taken during the second ether cook.

While the MiniCan^{TM} air samples captured 21 quantifiable VOCs during the One Pot methamphetamine cooks, the helium diffusion sampling personal monitors captured 11 quantifiable VOCs during the cooks. A summary of the VOCs captured by the 8 body-worn HDS personal monitors and their observed concentrations in ppb are shown in Table 10. Each HDS personal monitor air sample was given a name that follows the following template: Day-Cook-Person, so sample D1-C1-P1 means the sample was collected on the first day, during the first cook, and researcher 1 had the sampler clipped to their SCBA harness.

Table 10. Summary of the quantifiable VOCs collected with the 8 HDS personal monitors. All concentrations are in ppb air.

Cook	**Ether 1**		**Camp Fuel 1**		**Fail 1/Camp Fuel 2**		**Ether 2**	
Description (Day, Cook, Person)	D1-C1-P1	D1-C1-P2	D1-C2-P1	D1-C2-P2	D2-C1-P1	D2-C1-P2	D2-C2-P1	D2-C2-P2
Acetone	141.60	87.86	-	-	-	-	274.31	179.55
Ethyl Ether	2840.26	2007.51	-	-	-	-	2784.69	4184.51
Heptane	1054.92	765.84	272.21	268.95	963.00	1298.88	884.45	1574.39
2-Methyl butane	-	-	4026.53	4845.56	4572.00	6784.80	-	-
Pentane	-	-	128.87	156.26	164.25	258.72	-	-
Cyclohexane	-	-	835.03	865.44	2054.25	2985.84	-	-
Trichloroethene	-	-	-	162.27	1420.88	517.44	-	-
Isopropyl Alcohol	-	-	-	-	68.63	-	-	-
Methylene Chloride	-	-	-	-	419.63	150.48	-	-
Hexane	-	-	-	-	131.63	-	-	-
Methyl cyclohexane	-	-	-	-	88.88	-	-	-

A trend that can be observed in the data from Table 10, as well as from the gas chromatograms in Figures 30-33, is that for three of the four One Pot methamphetamine cooks, one researcher's HDS personal monitor captured a significantly greater amount of VOCs than the other researcher's monitor. For the first ether cook (Figure 30), researcher 1 had a greater exposure to VOCs and for the second ether cook (Figure 33) and the first camp fuel cook (Figure 31), researcher 2 had a greater exposure. During the cooks, one researcher added a majority of the chemicals to the One Pot while the other researcher prepped items that would be needed later in the cook and occasionally performed a burp. It is likely that researcher 1 in the first ether cook and researcher 2 in the second ether cook and first camp fuel cook were the researchers who did the chemical additions, as they would have been in direct contact with the One Pot throughout the cook, and many times they were leaning over the active One Pot during addition of sodium hydroxide, putting the HDS personal monitor very close to the open One Pot reaction vessel. During the first bottle failure and the second camp fuel cook (Figure 32), the HDS personal monitors worn by both researchers collected a similar amount of VOCs. This is likely due to the bottle failure spraying organic solvent throughout the cook shed and the researchers remaining in the shed during the second cook, allowing the HDS personal monitors to collect the VOCs from the expelled solvent as it evaporated.

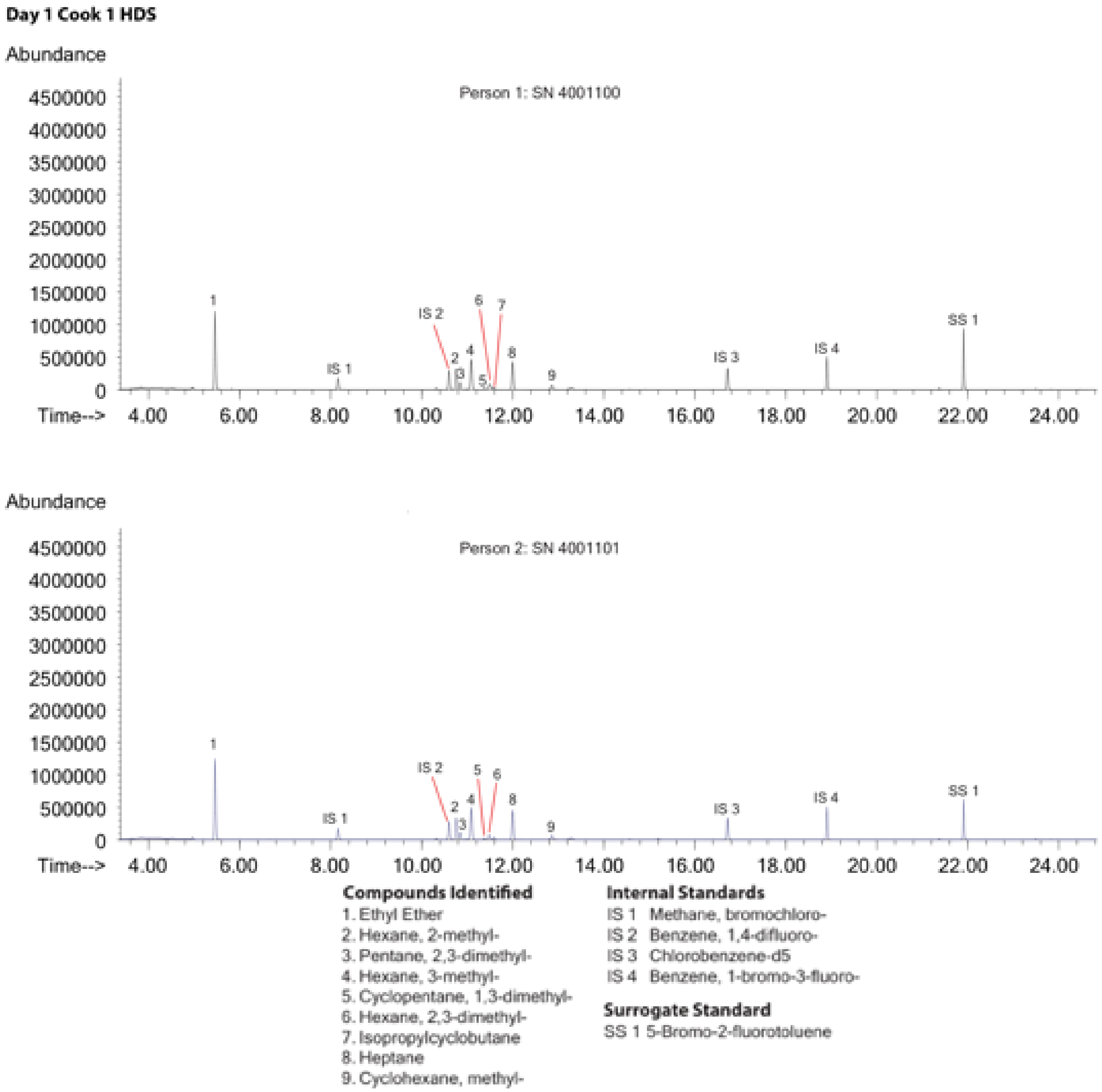


Figure 30. Gas chromatograms from the 2 HDS personal air samplers worn during the first ether cook, one sampler clipped to SCBA gear of each researcher.

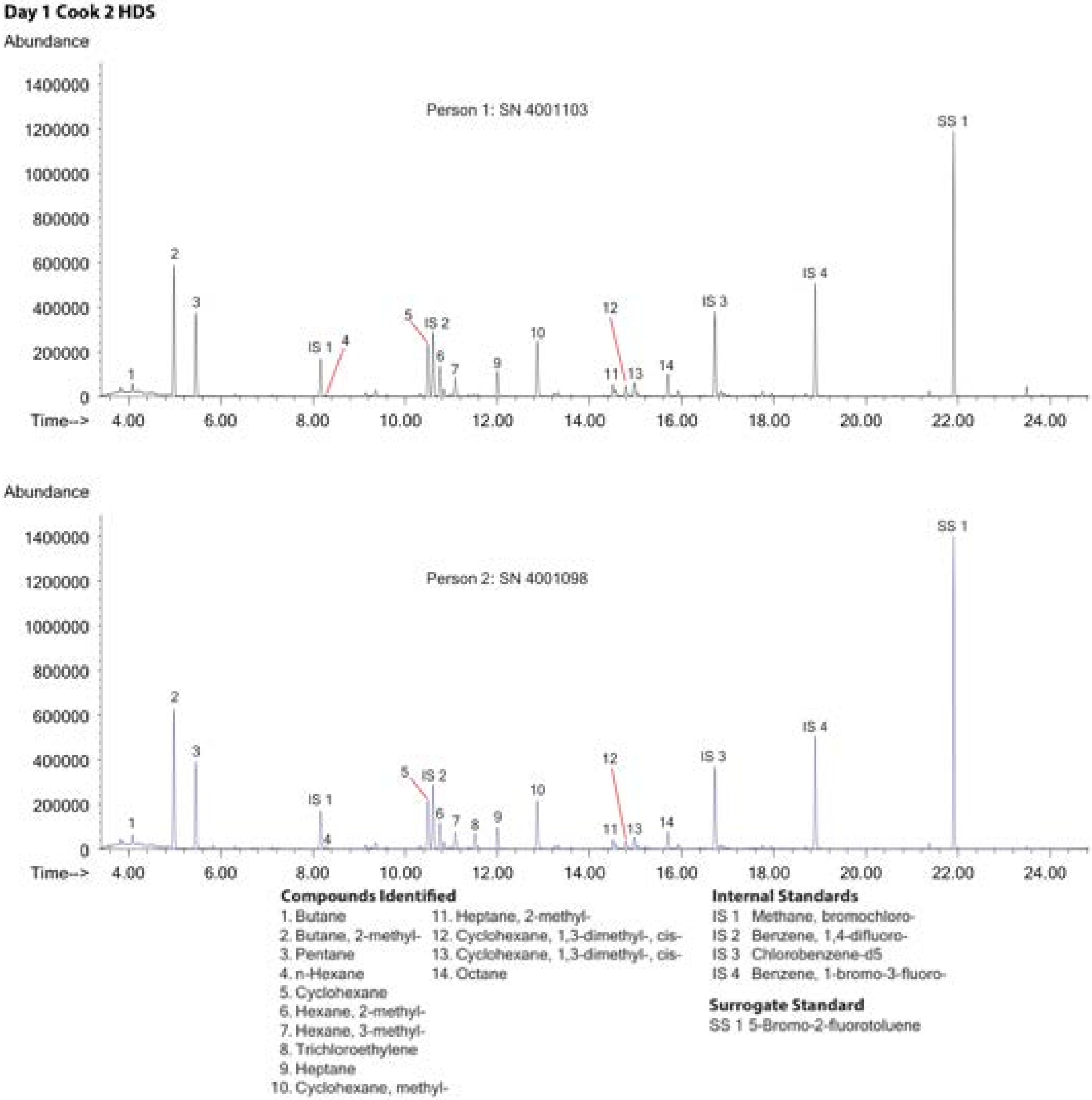


Figure 31. Gas chromatograms from the 2 HDS personal air samplers worn during the first camp fuel cook, one sampler clipped to SCBA gear of each researcher.

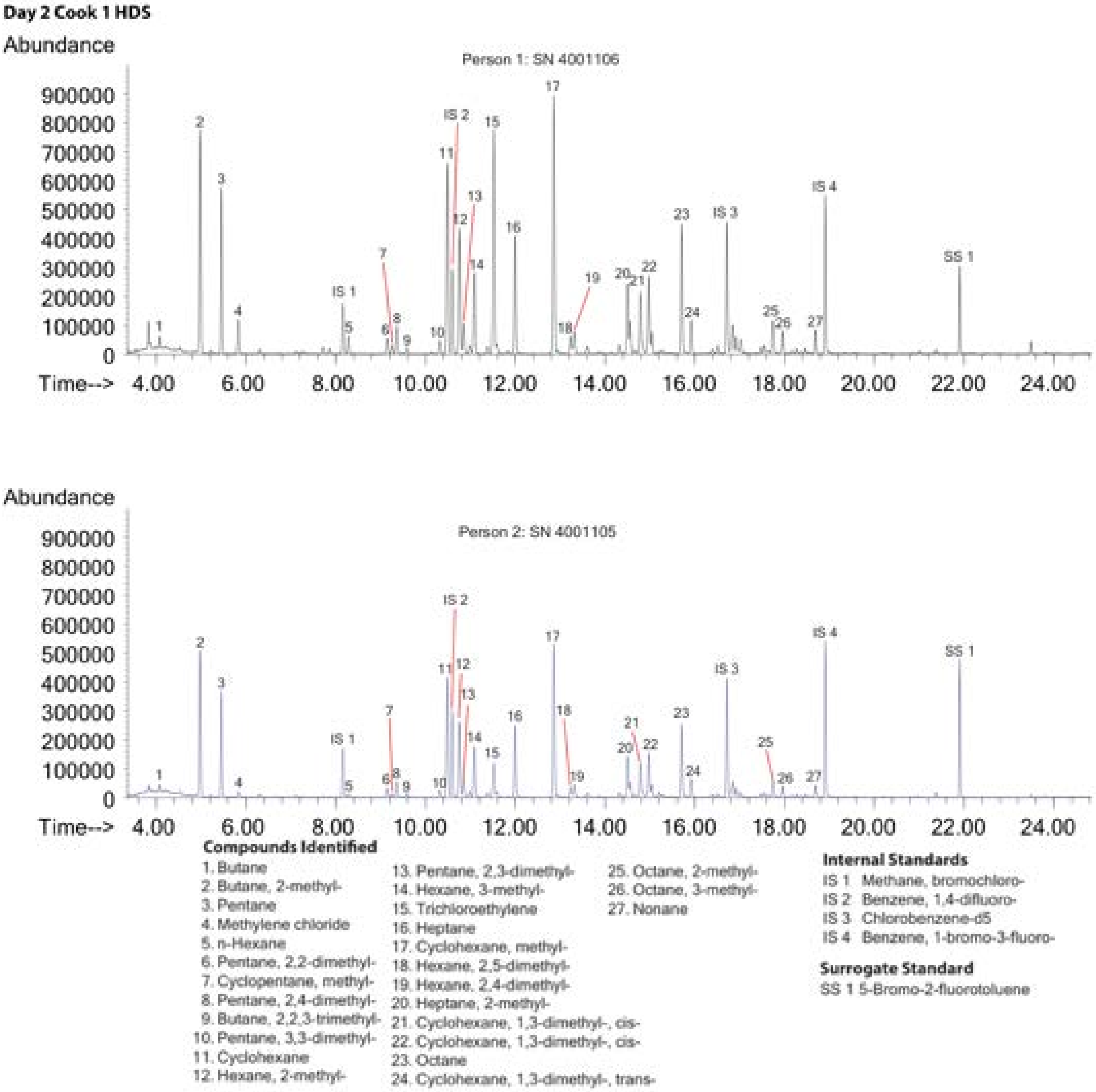


Figure 32. Gas chromatograms from the 2 HDS personal air samplers worn throughout the first bottle failure and the second camp fuel cook, one sampler clipped to SCBA gear of each researcher.

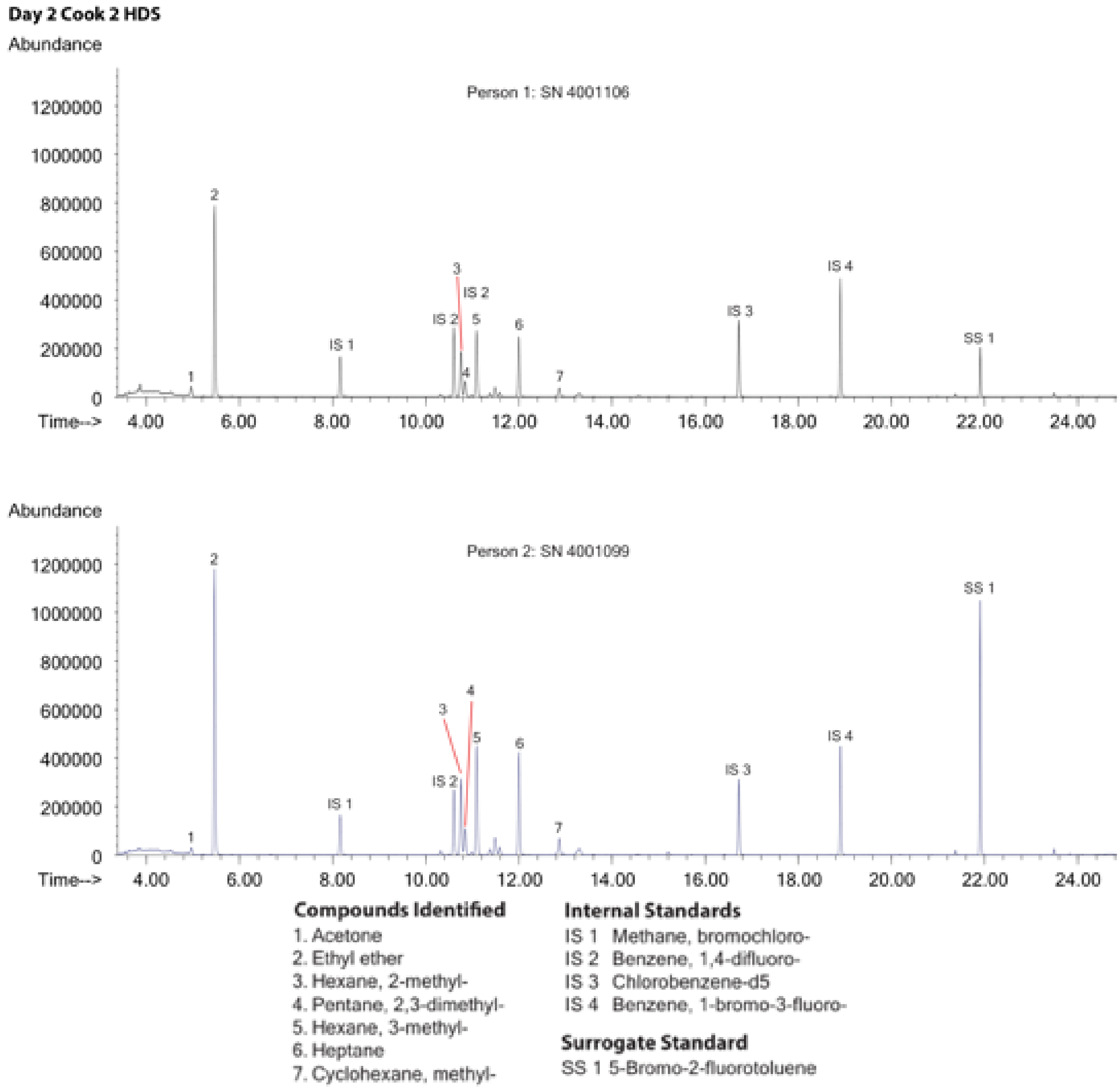


Figure 33. Gas chromatograms from the 2 HDS personal air samplers worn during the second ether cook, one sampler clipped to SCBA gear of each researcher.

While the sorbents in the MiniCans^{TM} and HDS personal monitors are designed to capture a wide range of VOCs, the Carbo X sorbent from the DSPs was designed to be more specific and better capture BTEX compounds. Due to this, quantitative analysis of these DSPs was only focused on BTEX compounds. These results are summarized in Table 11. As can be seen in this table, the only BTEX compounds with quantifiable concentrations were toluene and benzene. Quantified toluene and benzene concentrations follow the same trend as was observed with the HDS monitors, in that one researcher's DSP had collected a higher concentration of the BTEX compounds than the other researcher's, except in the case of the camp fuel failure, in which case both DSPs captured similar concentrations of BTEX compounds. One difference between the HDS and DSP results was that for the first camp fuel cook, the HDS found that Researcher Two had a greater exposure to the VOCs during the One Pot methamphetamine cook while the DSP found that Researcher One had a greater exposure. A likely explanation for this is that either the HDS or DSPs were switched between the two researchers, resulting in the conflicting data.

Table 11. Summary of the quantifiable BTEX compounds collected with the 8 DSP monitors. All concentrations are in ppb air.

Cook	**Ether 1**		**Camp Fuel 1**		**Fail 1/Camp Fuel 2**		**Ether 2**	
Description (Day, Cook, Person)	D1-C1-P1	D1-C1-P2	D1-C2-P1	D1-C2-P2	D2-C1-P1	D2-C1-P2	D2-C2-P1	D2-C2-P2
Toluene	0.81	-	0.24	-	0.30	0.26	-	0.23
Benzene	-	-	0.27	-	-	-	-	0.33

Though the Carbo X sorbent in the DSPs is designed to capture BTEX compounds, other compounds are also captured with these passive air sampling devices. This can be seen by observing the gas chromatograms associated with these samplers in Figures 34-37.

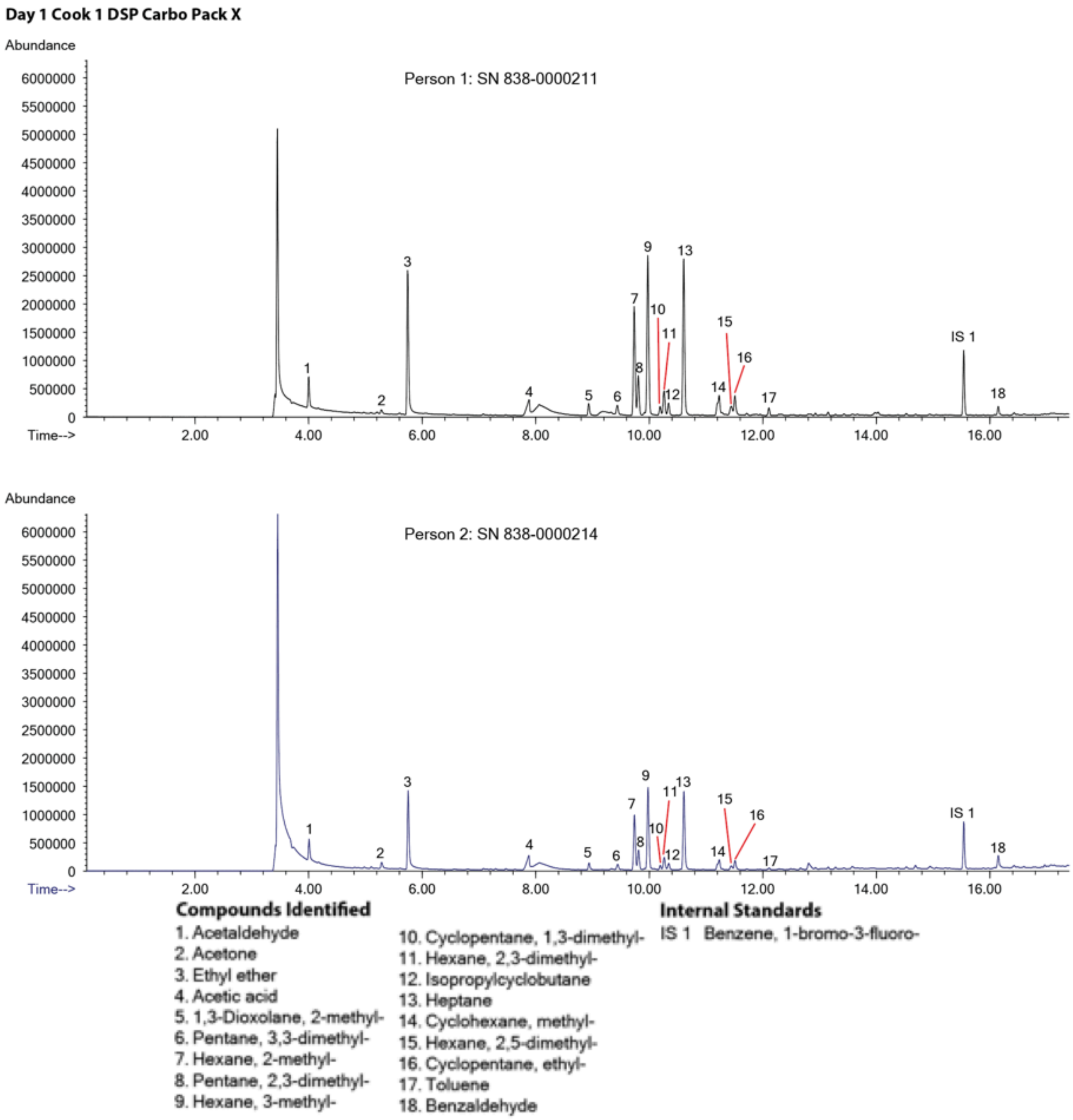


Figure 34. Gas chromatograms from the 2 Carbo X DSP samplers worn during the first ether cook.

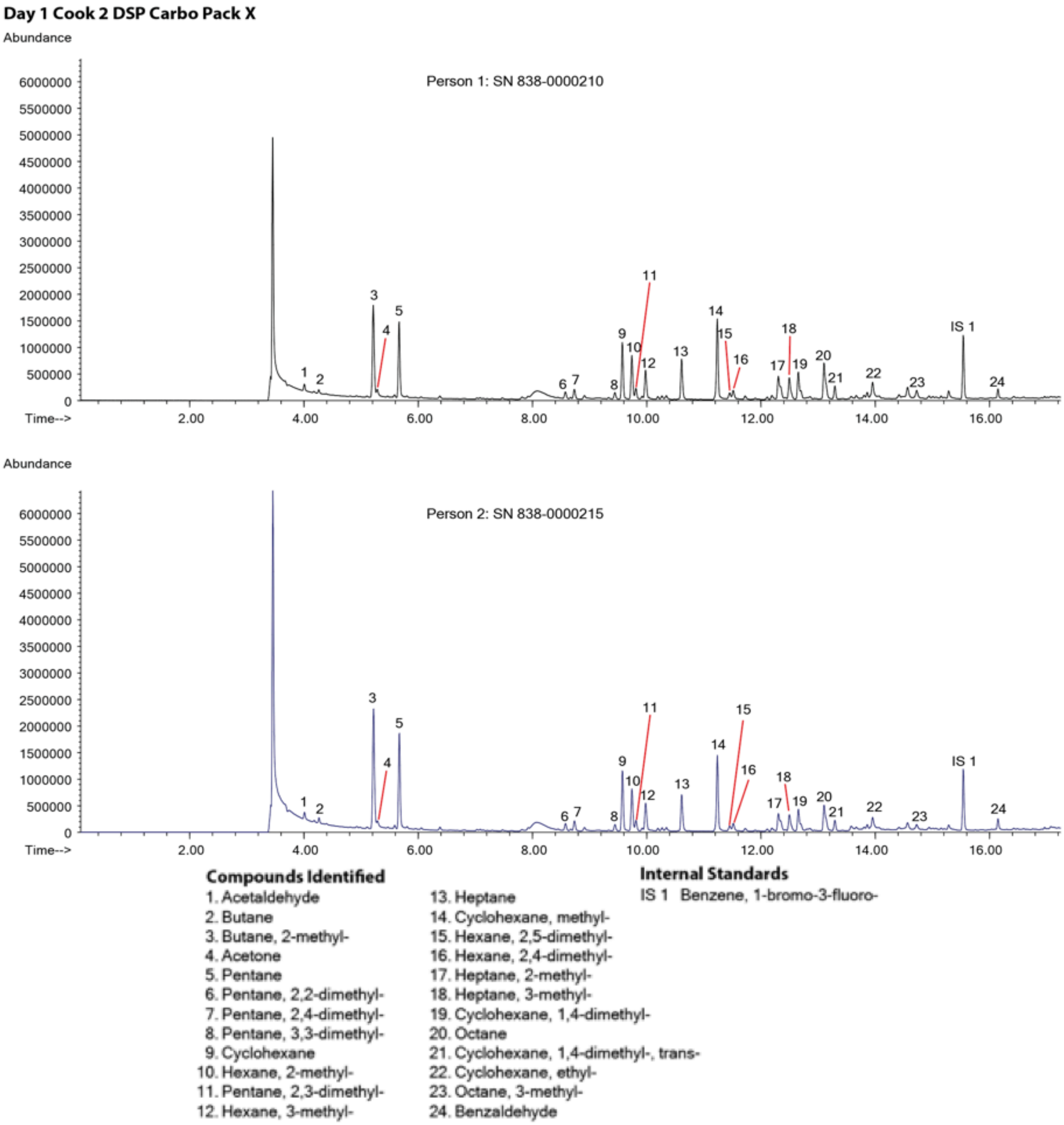
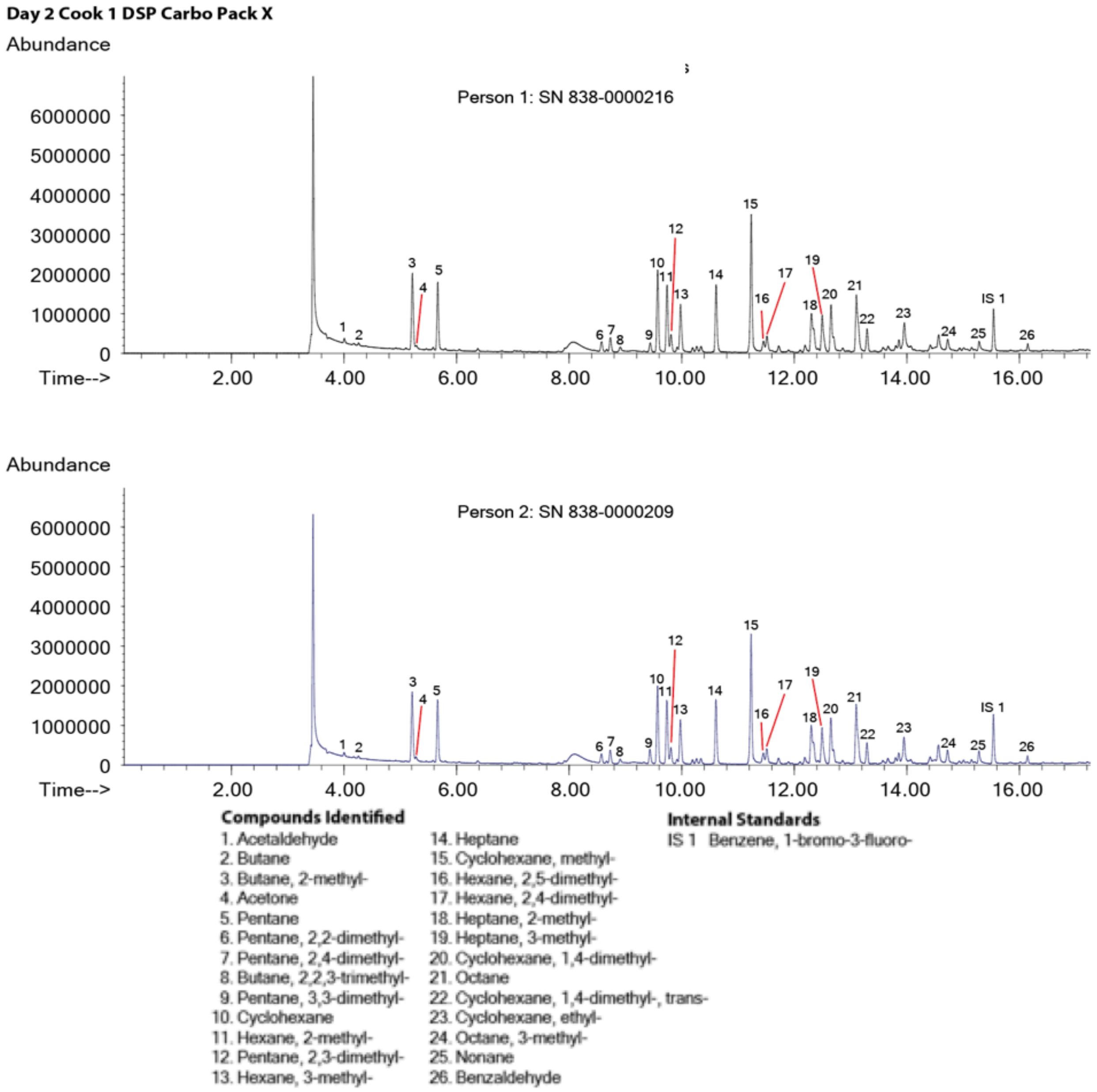


Figure 35. Gas chromatograms from the 2 Carbo X DSP samplers worn during the first camp fuel cook.



Day 2 Cook 1 DSP Carbo Pack X

Figure 36. Gas chromatograms from the 2 Carbo X DSP samplers worn during the first bottle failure and second camp fuel cook.

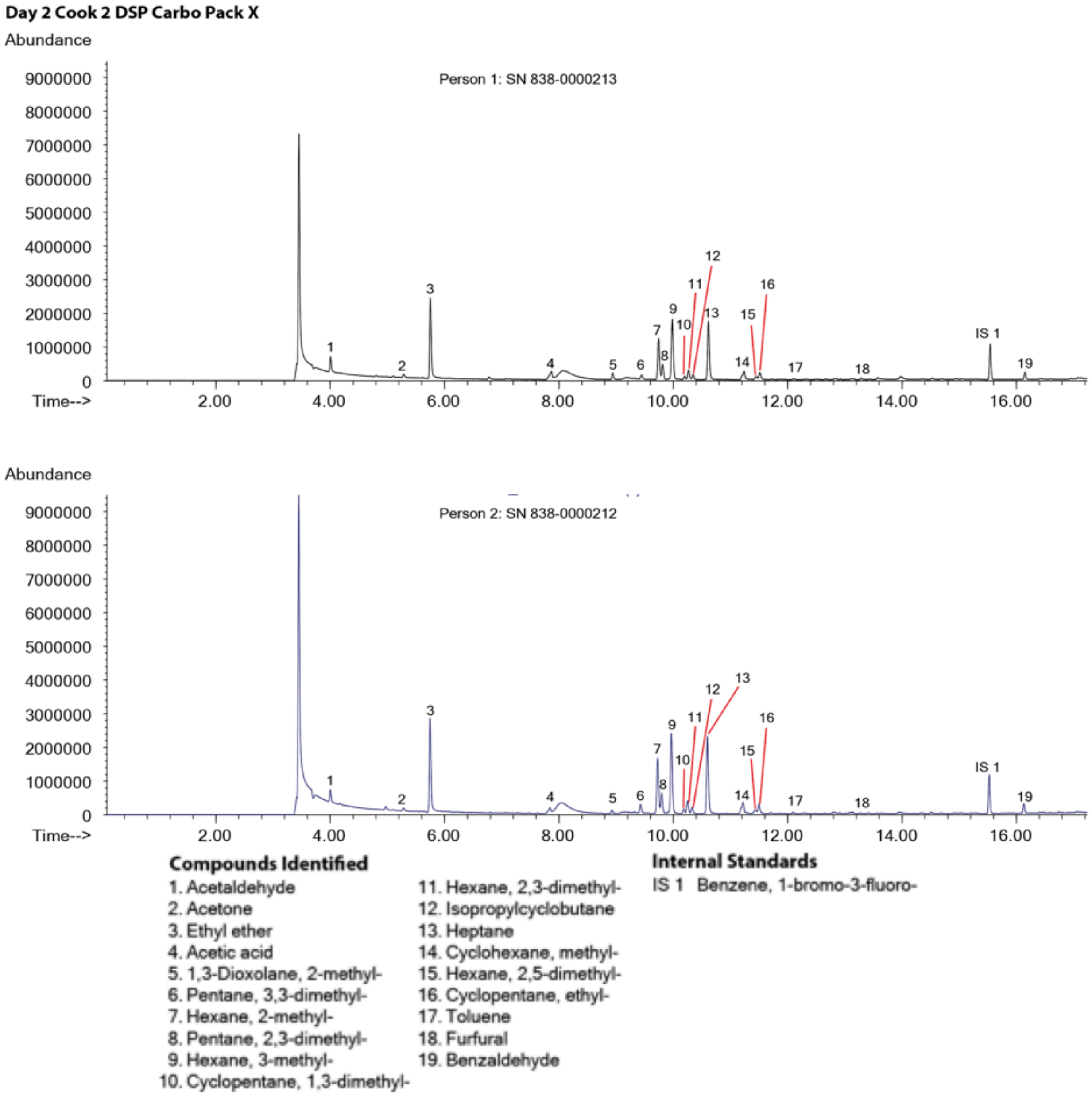
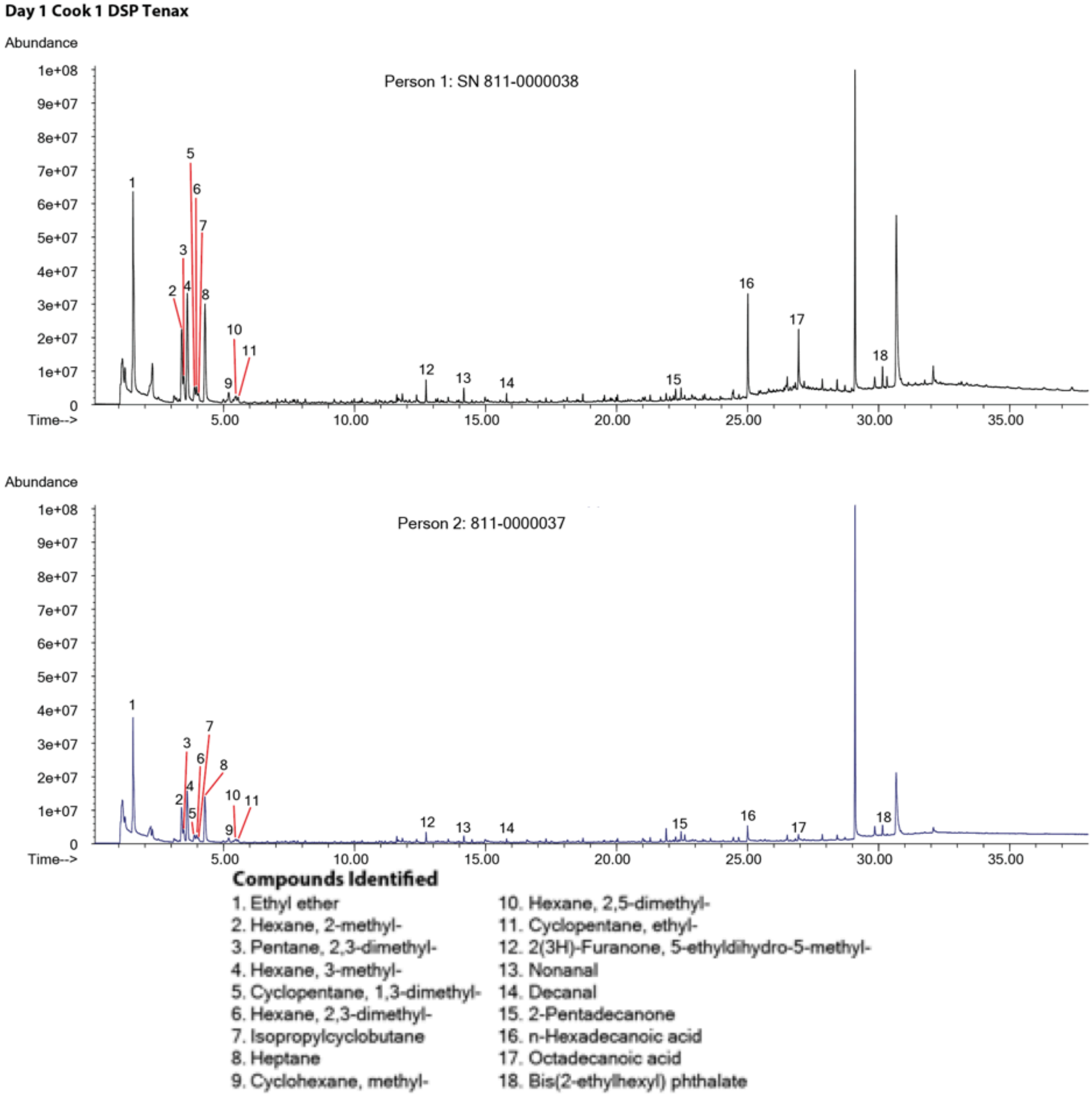


Figure 37. Gas chromatograms from the 2 Carbo X DSP samplers worn during the second ether cook.

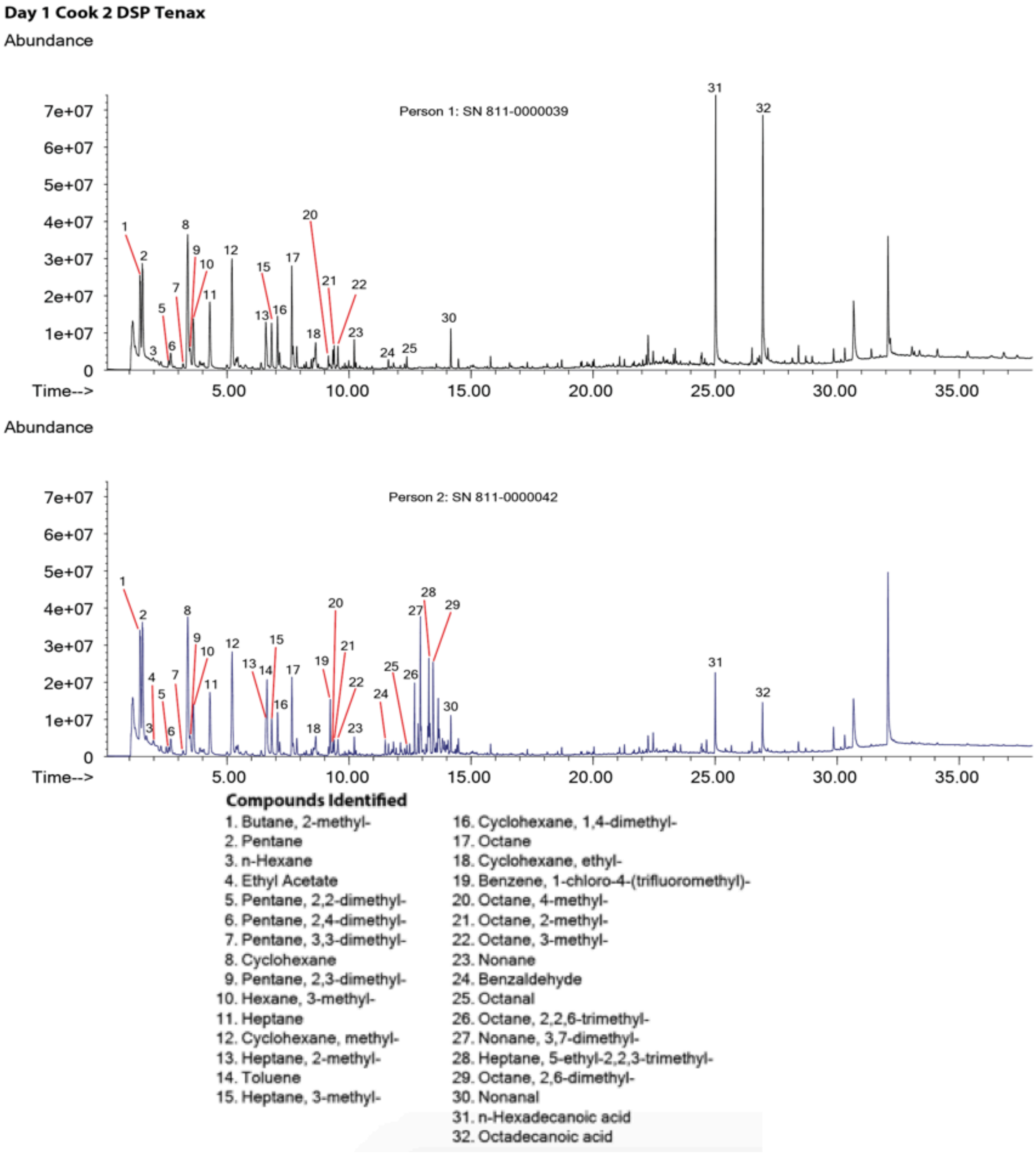
While the Carbo X DSPs are optimal for the collection and analysis of BTEX compound, the Tenax DSPs are designed more for the collection and analysis of VOCs with higher boiling points, such as long-chained hydrocarbons. The Tenax DSPs used during this study were used solely in a qualitative manner. The identified chromatograms associated with these Tenax DSPs are shown in Figures 38-41.



Compounds Identified

1. Ethyl ether
2. Hexane, 2-methyl-
3. Pentane, 2,3-dimethyl-
4. Hexane, 3-methyl-
5. Cyclopentane, 1,3-dimethyl-
6. Hexane, 2,3-dimethyl-
7. Isopropylcyclobutane
8. Heptane
9. Cyclohexane, methyl-
10. Hexane, 2,5-dimethyl-
11. Cyclopentane, ethyl-
12. 2(3H)-Furanone, 5-ethyldihydro-5-methyl-
13. Nonanal
14. Decanal
15. 2-Pentadecanone
16. n-Hexadecanoic acid
17. Octadecanoic acid
18. Bis(2-ethylhexyl) phthalate

Figure 38. Gas chromatograms from the 2 Tenax DSP samplers worn during the first ether cook.



Compounds Identified

1. Butane, 2-methyl-
2. Pentane
3. n-Hexane
4. Ethyl Acetate
5. Pentane, 2,2-dimethyl-
6. Pentane, 2,4-dimethyl-
7. Pentane, 3,3-dimethyl-
8. Cyclohexane
9. Pentane, 2,3-dimethyl-
10. Hexane, 3-methyl-
11. Heptane
12. Cyclohexane, methyl-
13. Heptane, 2-methyl-
14. Toluene
15. Heptane, 3-methyl-
16. Cyclohexane, 1,4-dimethyl-
17. Octane
18. Cyclohexane, ethyl-
19. Benzene, 1-chloro-4-(trifluoromethyl)-
20. Octane, 4-methyl-
21. Octane, 2-methyl-
22. Octane, 3-methyl-
23. Nonane
24. Benzaldehyde
25. Octanal
26. Octane, 2,2,6-trimethyl-
27. Nonane, 3,7-dimethyl-
28. Heptane, 5-ethyl-2,2,3-trimethyl-
29. Octane, 2,6-dimethyl-
30. Nonanal
31. n-Hexadecanoic acid
32. Octadecanoic acid

Figure 39. Gas chromatograms from the 2 Tenax DSP samplers worn during the first camp fuel cook.

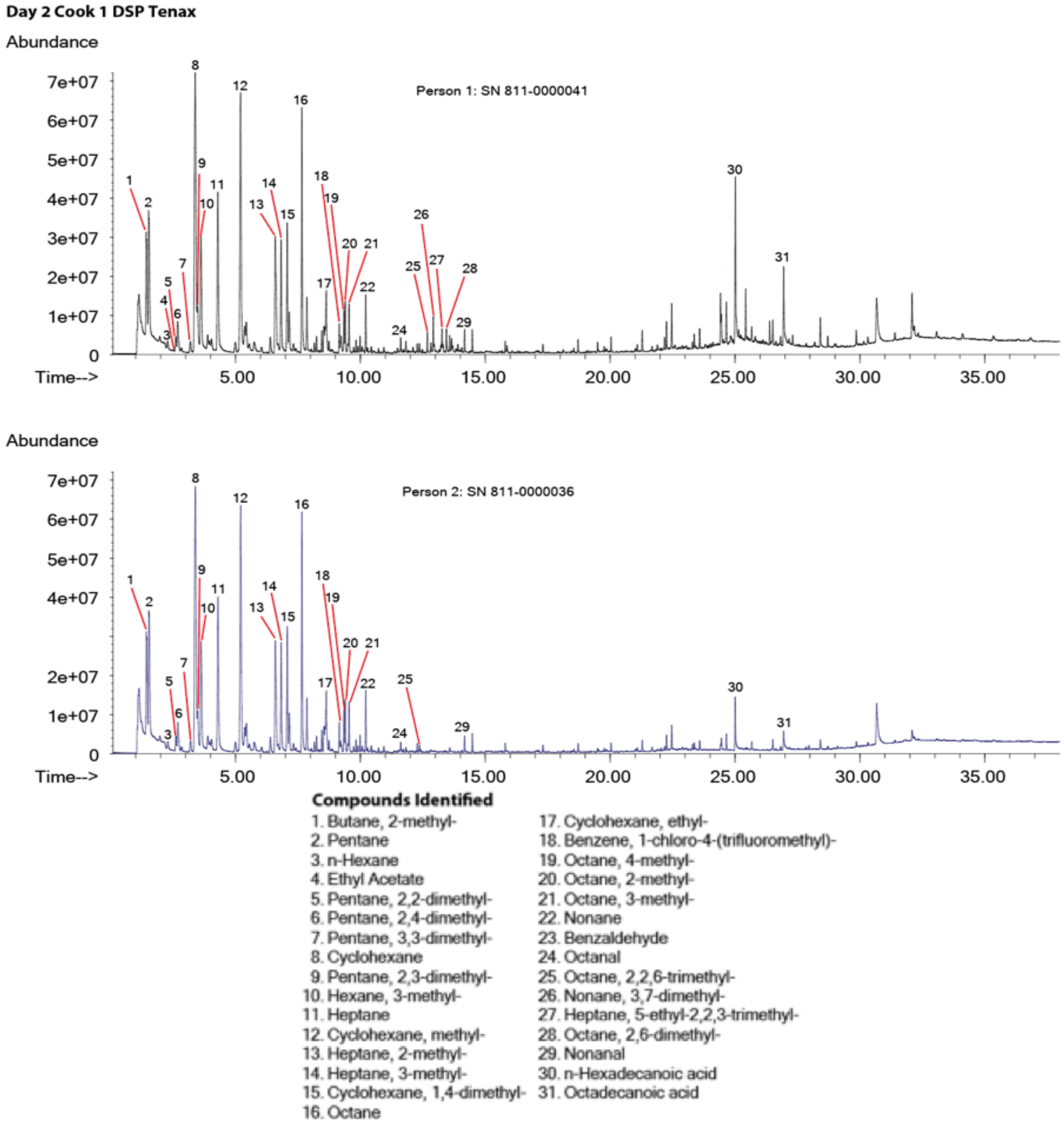

Day 2 Cook 1 DSP Tenax

Figure 40. Gas chromatograms from the 2 Tenax DSP samplers worn during the first bottle failure and second camp fuel cook.

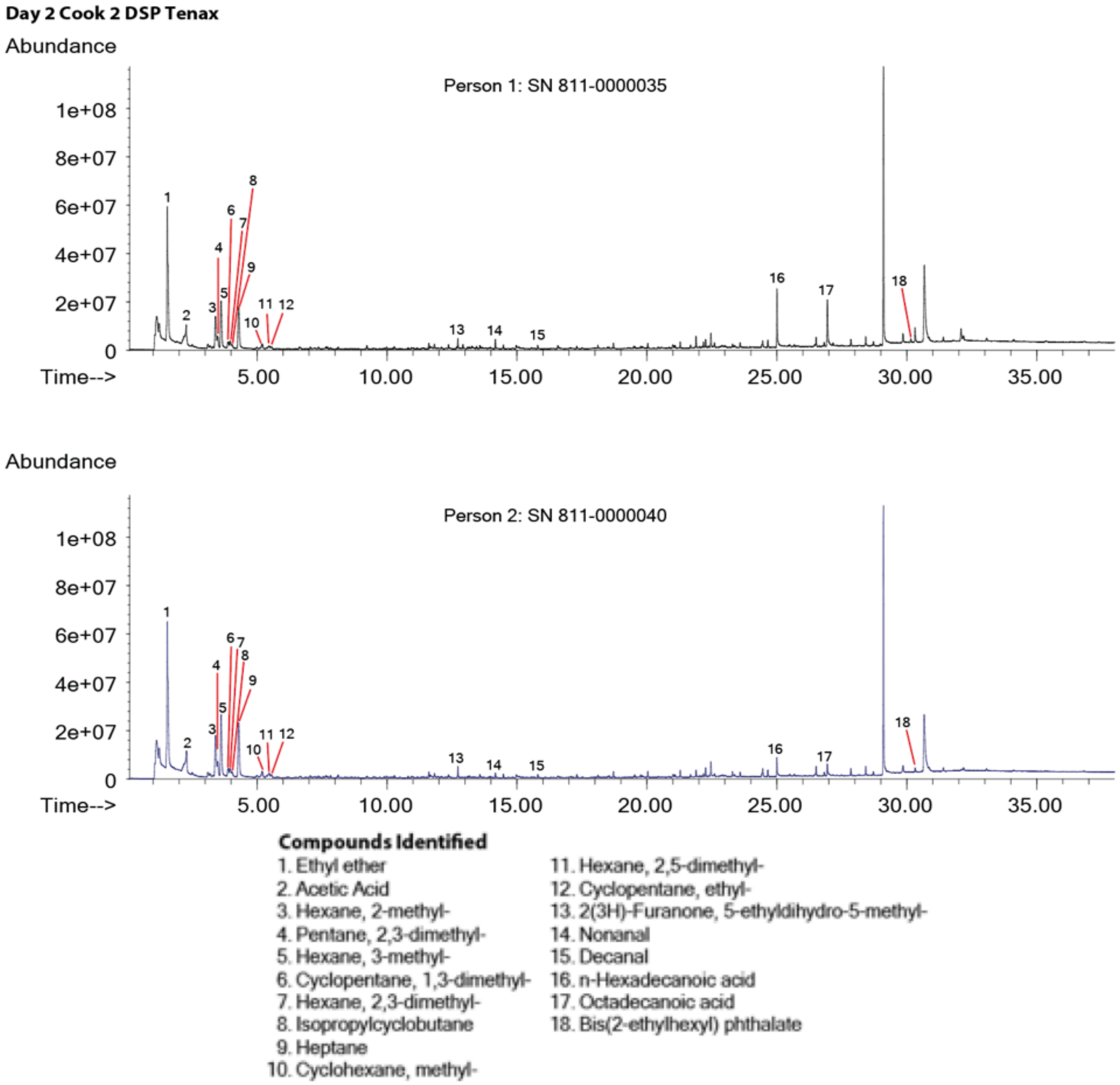
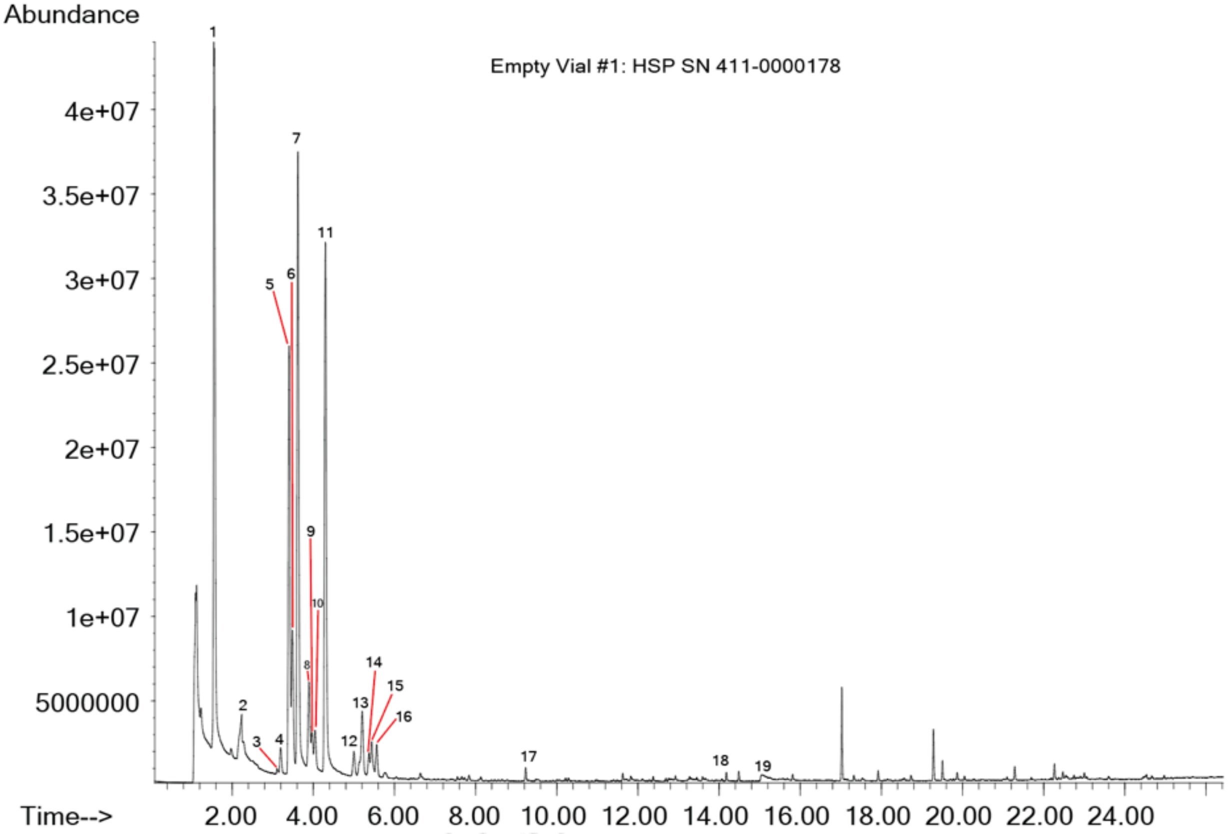


Figure 41. Gas chromatograms from the 2 Tenax DSP samplers worn during the second ether cook.

As noted above, the 40 mL screw-top vial grab sample was left opened to the environment just prior to assembling the HCl gas generator and was capped following filtration of the powdered methamphetamine from the post-salt solvent. As with the Tenax DSP, the 40 mL screw-top grab samples were analyzed as qualitative only. As these screw-top vials were left sitting open in the atmosphere, any VOCs that were in the air could settle into the vial, with those in present in the highest concentration most likely to fill the vial. The chromatograms for the 40 mL screw-top grab samples are shown in Figures 42-45.

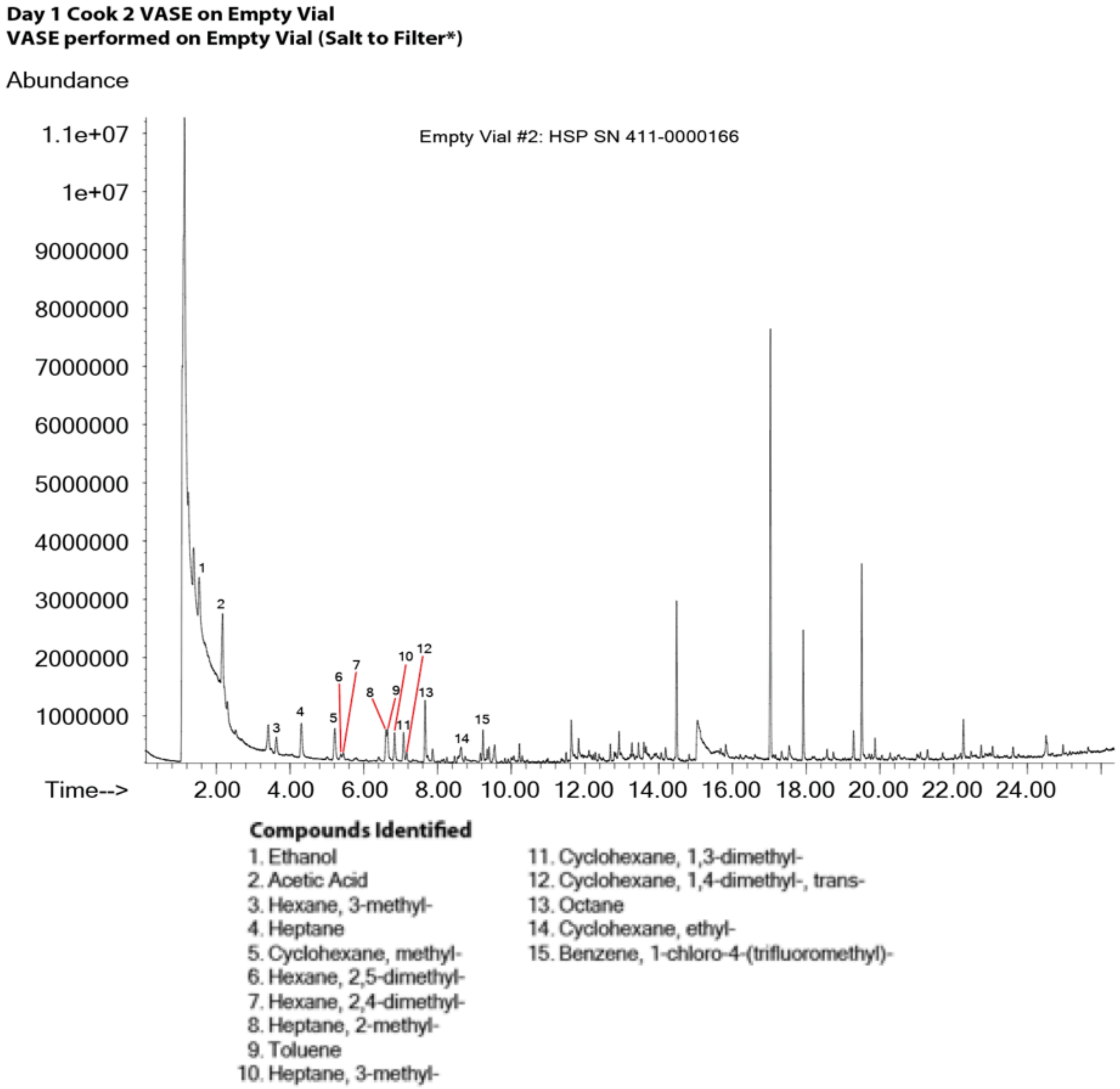
Day 1 Cook 1 VASE on Empty Vial
VASE performed on Empty Vial (Salt to Filter*)



Compounds Identified

1. Ethyl ether
2. Acetic Acid
3. 1,3-Dioxolane, 2-methyl-
4. Pentane, 3,3-dimethyl-
5. Hexane, 2-methyl-
6. Pentane, 2,3-dimethyl-
7. Hexane, 3-methyl-
8. Cyclopentane, 1,3-dimethyl-
9. Hexane, 2,3-dimethyl-
10. Isopropylcyclobutane
11. Heptane
12. Hexane, 2,2-dimethyl-
13. Cyclohexane, methyl-
14. Hexane, 2,5-dimethyl-
15. Hexane, 2,4-dimethyl-
16. Cyclopentane, ethyl-
17. Benzene, 1-chloro-4-(trifluoromethyl)-
18. Nonanal
19. Benzoic acid

Figure 42. Gas chromatogram from the 40 mL screw-top vial opened prior to salting out the first ether cook.



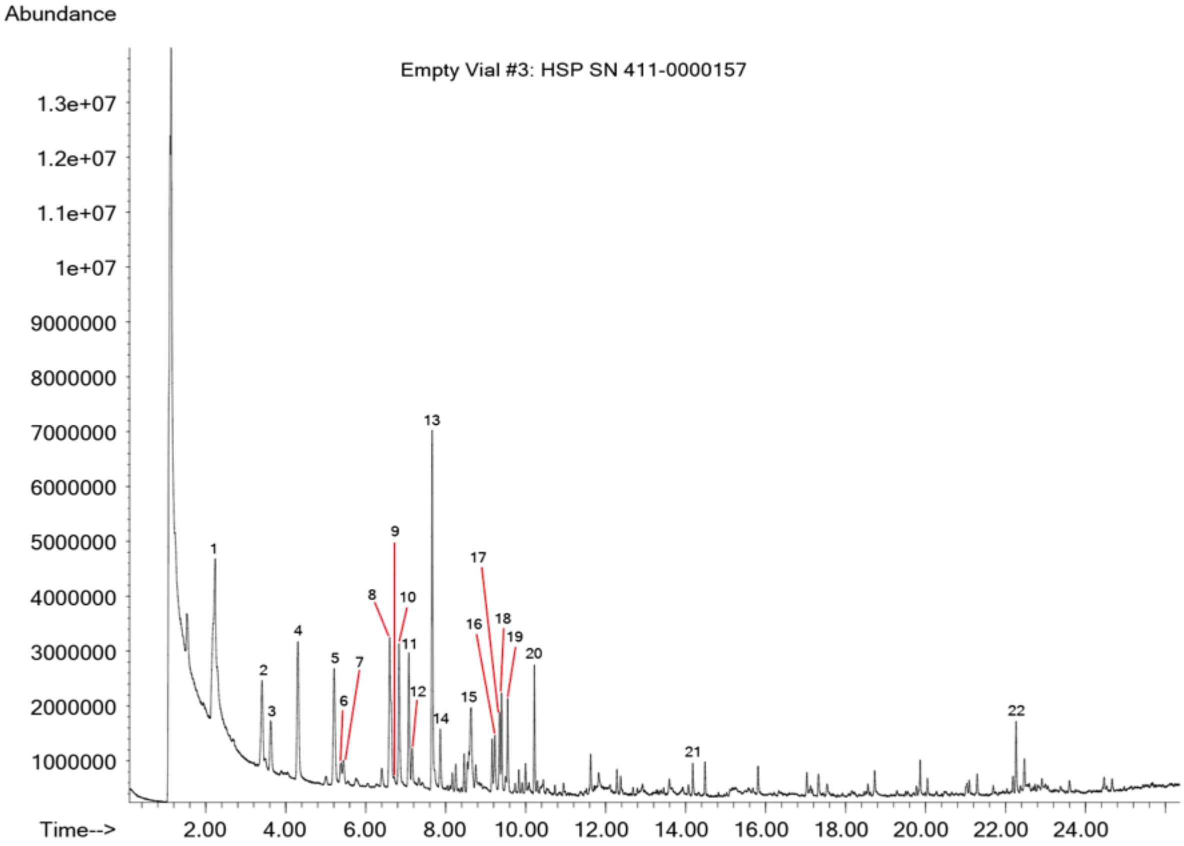
Day 1 Cook 2 VASE on Empty Vial
VASE performed on Empty Vial (Salt to Filter*)

Compounds Identified

1. Ethanol
2. Acetic Acid
3. Hexane, 3-methyl-
4. Heptane
5. Cyclohexane, methyl-
6. Hexane, 2,5-dimethyl-
7. Hexane, 2,4-dimethyl-
8. Heptane, 2-methyl-
9. Toluene
10. Heptane, 3-methyl-
11. Cyclohexane, 1,3-dimethyl-
12. Cyclohexane, 1,4-dimethyl-, trans-
13. Octane
14. Cyclohexane, ethyl-
15. Benzene, 1-chloro-4-(trifluoromethyl)-

Figure 43. Gas chromatogram from the 40 mL screw-top vial opened prior to salting out the first camp fuel cook.

Day 2 Cook 1 VASE on Empty Vial
VASE performed on Empty Vial (Salt to Filter*)



Compounds Identified

1. Acetic Acid
2. Hexane, 2-methyl-
3. Hexane, 3-methyl-
4. Heptane
5. Cyclohexane, methyl-
6. Hexane, 2,5-dimethyl-
7. Hexane, 2,4-dimethyl-
8. Heptane, 2-methyl-
9. Toluene
10. Heptane, 3-methyl-
11. Cyclohexane, 1,4-dimethyl-
12. Cyclohexane, 1,4-dimethyl-, trans-
13. Octane
14. Cyclohexane, 1,3-dimethyl-
15. Cyclohexane, ethyl-
16. Benzene, 1-chloro-4-(trifluoromethyl)-
17. Octane, 4-methyl-
18. Octane, 2-methyl-
19. Octane, 3-methyl-
20. Nonane
21. Nonanal
22. Hexadecane

Figure 44. Gas chromatogram from the 40 mL screw-top vial opened prior to salting out the second camp fuel cook.

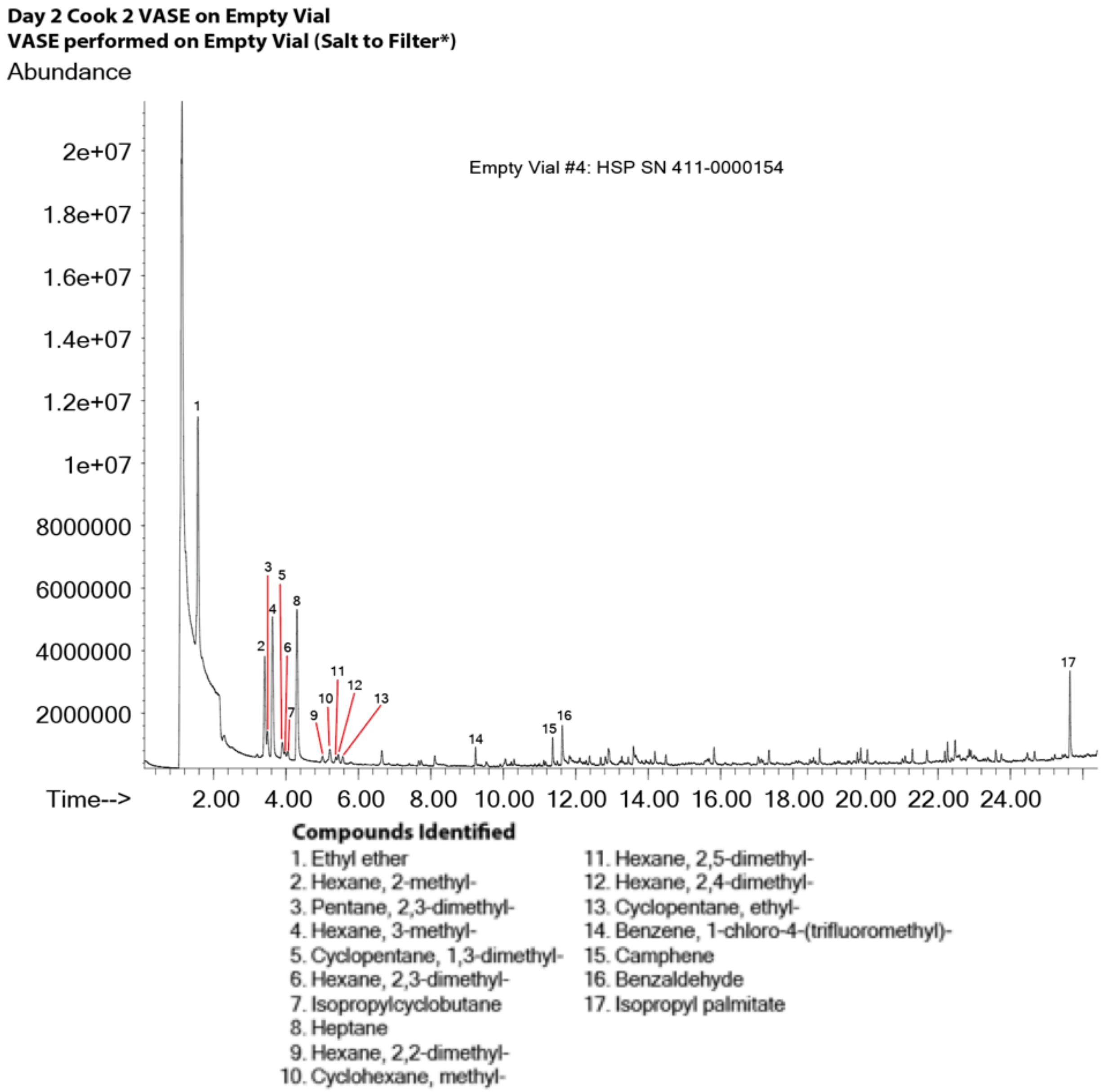


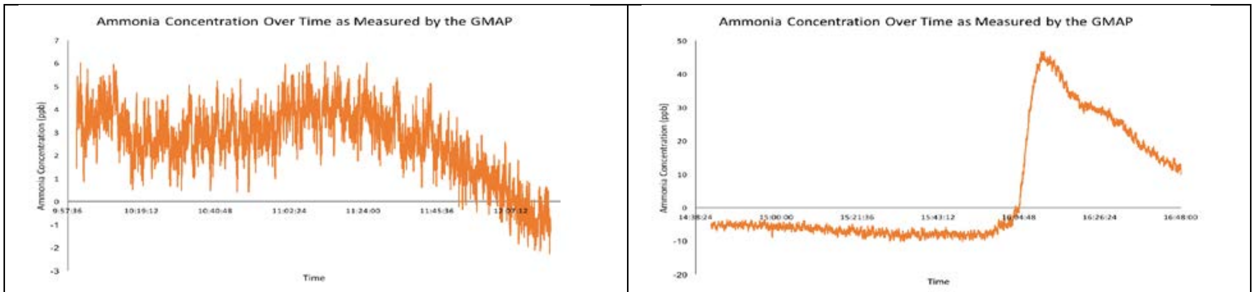
Figure 45. Gas chromatogram from the 40 mL screw-top vial opened prior to salting out the second ether cook.

Standoff Air Monitoring

Ammonia Detection

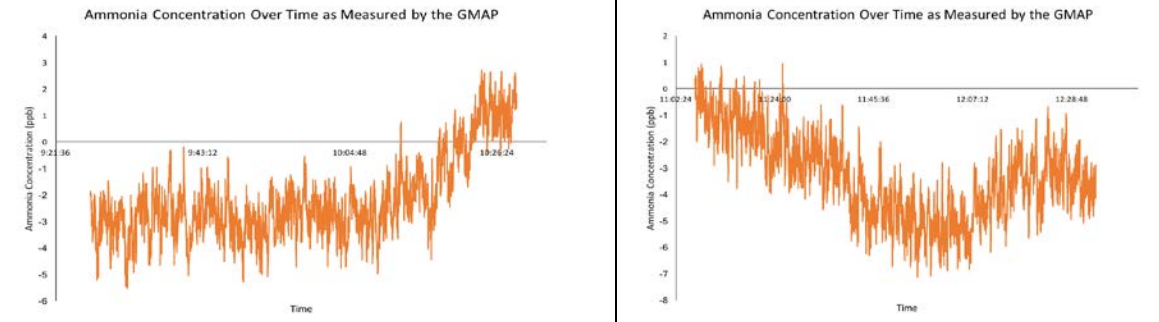
The GMAP unit on-board differential ultraviolet absorption spectrometer (DUVAS) and photoionization detector (PID) were utilized for detection of ammonia and VOCs respectively, but since methane and carbon dioxide were not of interest for this project, the integrated cavity output spectroscopy (ICOS) detector also in the vehicle instrument deck was not utilized. While the DUVAS was designed for analysis of benzene, toluene, ethylbenzene, and xylenes, this study utilized the DUVAS for analysis of ammonia that was produced during the One Pot methamphetamine cooks. Using the DUVAS for ammonia detection was not ideal, as the DUVAS was not calibrated in the field for ammonia prior to collecting samples, the DUVAS varied from standard ammonia sampling practices by not having a heated sampling line, and the stainless steel fittings on the DUVAS sampling line were not designed for ammonia analysis, as they provide absorption sites for the polar ammonia molecules to adhere to. Due to these limitations in the ammonia measurement system, all ammonia measurements by DUVAS are considered qualitative only. Also, the time when the DUVAS began collecting data may in some cases have occurred prior to or after the beginning of the One Pot methamphetamine cook.

Figure 46 shows the ammonia concentrations measured by the DUVAS for all eight One Pot events. During the One Pot cooks, the GMAP was parked about 15 meters from the cook shed and during the fire control study, the GMAP was parked 30 meters from the burn site. Due to the DUVAS not being optimized for ammonia detection, the data shown is qualitative only. This lack of optimization is observed in Figures 46 b-h, which have a negative ammonia concentration as a baseline. For reference purposes, previous research has determined Oklahoma to have an average ambient gaseous ammonia concentration of 1.8 ppb.[15] While the concentration of ammonia cannot be determined with the DUVAS used in this study, the change in ammonia concentration can still be visualized with the data. Although the in-vehicle DUVAS was located immediately adjacent to the cook shed, only Figures 46 b, f, and g present a clear ammonia detection signal, suggesting even at 15 meters from the shed, plume detection is very dependent on wind direction, with Figures 46 a, c, d, e, and h possibly depicting instrument drift at the noise floor of the detector.



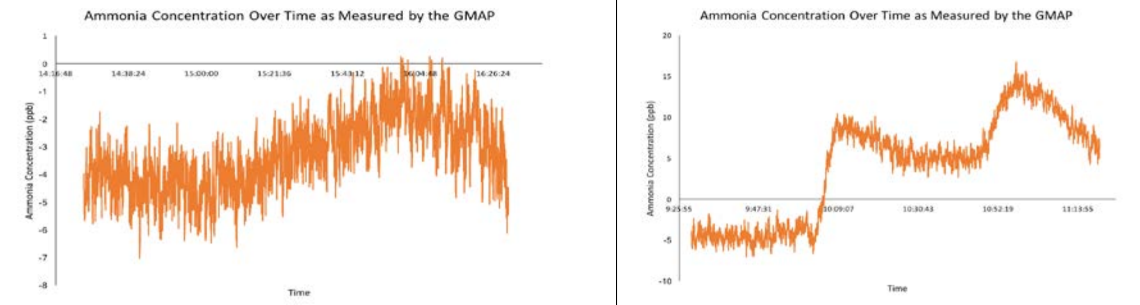
a) First ether cook

b) First camp fuel cook



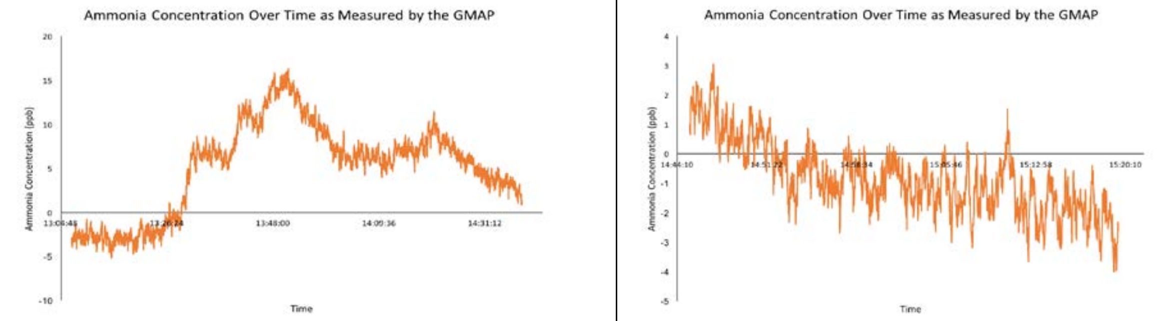
c) First bottle failure

d) Second camp fuel cook



e) Second ether cook

f) Second bottle failure



g) Third ether cook

h) Fire Control study

Figure 46. Ammonia concentrations measured by the DUVAS at 15m during each One Pot cook and 30m during the fire control study.

While the DUVAS was ineffective in collecting ammonia plume data during the first ether cook, the first bottle failure, the second camp fuel cook, the second ether cook, and the fire control study (Figures 46 a, c, d, e and h), it was successful in collecting this data during the first camp fuel cook (Figure 47), the second bottle failure (Figure 48), and the third ether cook (Figure 49). In Figure 47, the concentration of ammonia gas observed was stable from 14:38-16:04. At 16:04 the One Pot was filtered, releasing the ammonia gas from the cook into the atmosphere. This plume of ammonia gas was readily observed by the DUVAS, and the gradual decline in ammonia gas concentration can be observed as the ammonia diffused out of the shed and into the surrounding environment.

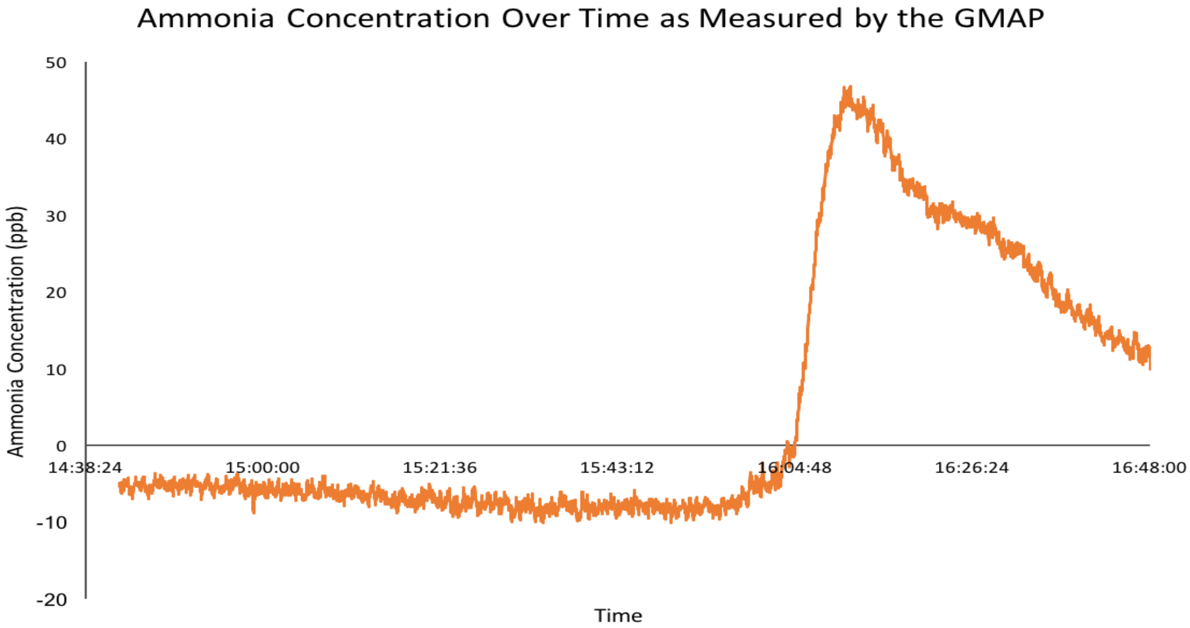


Figure 47. Ammonia concentrations measured by the DUVAS during the first camp fuel One Pot methamphetamine cook.

Figure 48 shows the ammonia concentration measured by the DUVAS during the second bottle failure. In this figure, two separate increases in ammonia concentration can be observed. The first increase occurred around 10:00 and corresponds to the actual bottle failure event. A slight decrease in ammonia concentration is then observed as the contents of the failed One Pot were added to a plastic food saver and the One Pot cook was completed as the ammonia from the bottle failure diffused throughout the atmosphere. The second increase in ammonia concentration occurred around 10:52, which was when the lid was removed from the plastic food saver so the One Pot could be filtered.

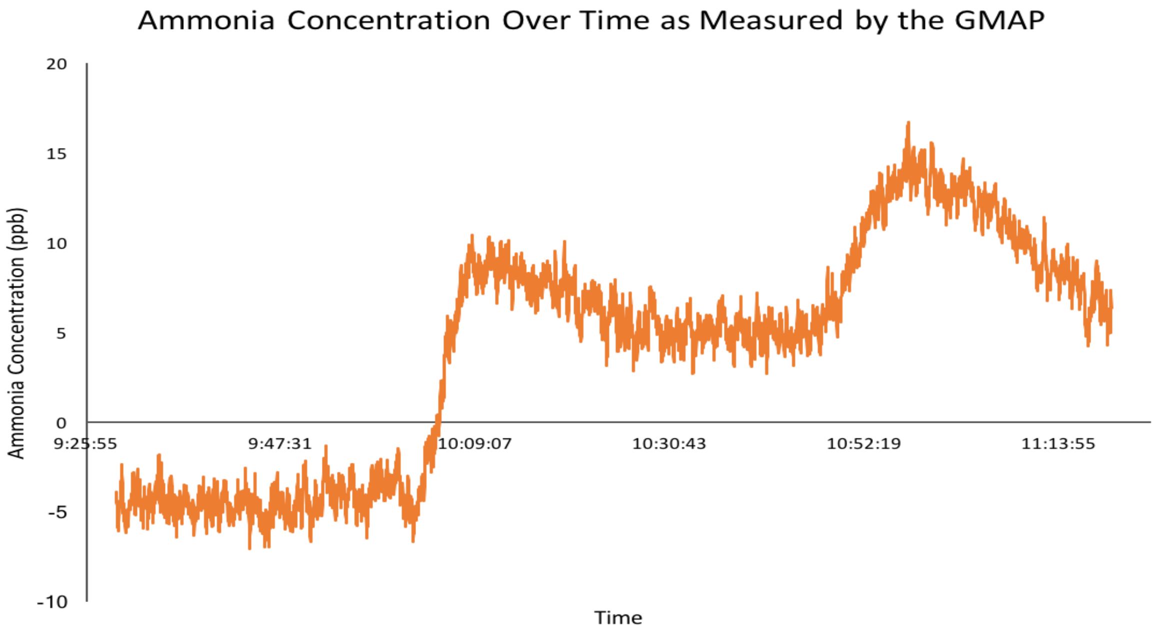


Figure 48. Ammonia concentrations measured by the DUVAS during the second bottle failure.

Figure 49 shows the ammonia concentration measured by the DUVAS during the third ether One Pot methamphetamine cook. Four increases in ammonia concentration were observed during the cook around 13:28, 13:38, 13:43, and 14:15. The first three increases in ammonia were a result of burping the One Pot. The third ether cook was the only cook in which the release of ammonia gas during burping could be observed. This is likely due to negligible winds and the optimal positioning of the GMAP unit with respect to the cook shed and wind direction. What can be observed during the burping events is an additive effect of burping on ammonia concentration. In other words, more ammonia was released during each burping event then could be diffused out of the shed between burping events, causing the concentration of ammonia gas to continue to grow until the wind speed increased and changed direction after the 13:43 burping event. The ammonia concentration then decreased as the ammonia diffused into the environment, and around 14:15 the One Pot reaction vessel was opened for filtration, causing a final increase in the observed ammonia concentration.

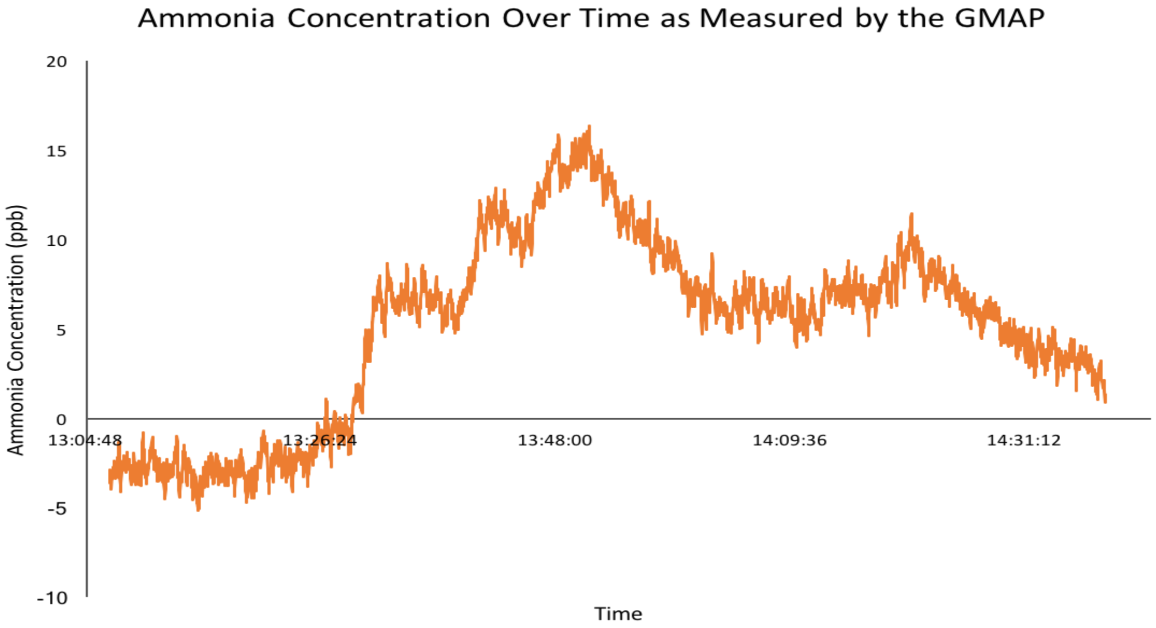


Figure 49. Ammonia concentrations measured by the DUVAS during the third ether One Pot methamphetamine cook.

Figures 50-56 chart ammonia concentrations measured by the ICOS Gas Analyzer during 5 of the cooks and the fire control study at various standoff distances as noted. Burp spikes are most clearly noted at 40 m standoff (See Figure 57). Driving loops varied from slow speeds of 3-5 mph to a maximum of 10-20 mph. The distinctive “meth lab smell” attributed to ammonia was noticeable intermittently, occurring at standoff distances as far as 150 meters. During the active Thursday afternoon ether cook, the distinctive odor was noticed while driving at approximately 75 m from the shed in a 100 m loop, even in the absence of an evident ammonia signal, leading to a shortened driving circuit of 45m and improved ammonia signal, as seen in Figure 55. ICOS distance from cook shed and field notes summarized in Table 12.

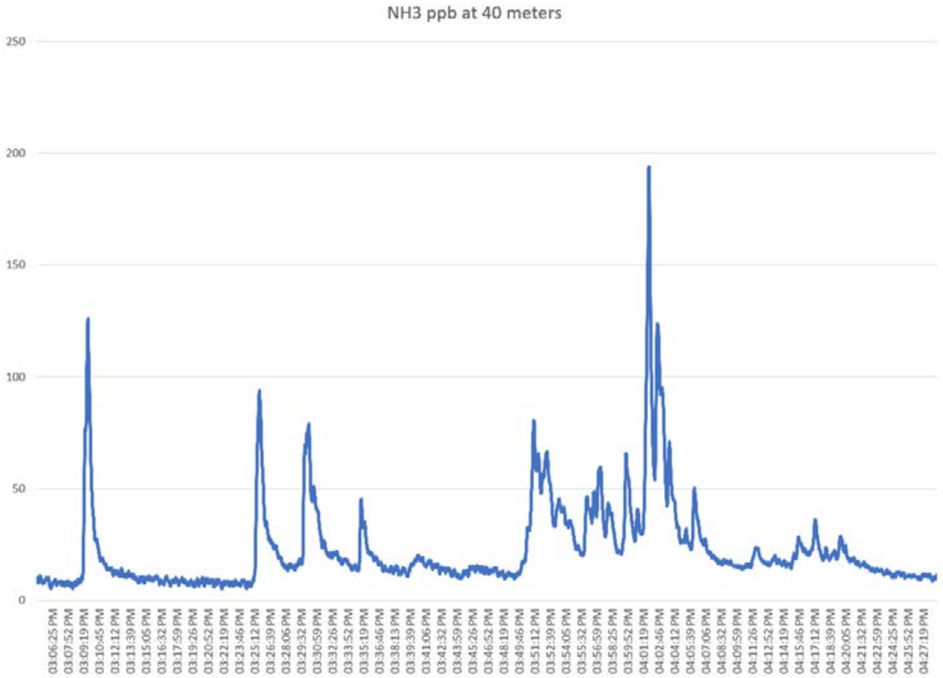


Figure 50. ICOS ammonia readings taken during the Tuesday afternoon camp fuel cook at 40 meters from the cook shed. One Pot burps are evident at 5 minute intervals beginning 3:25 pm.

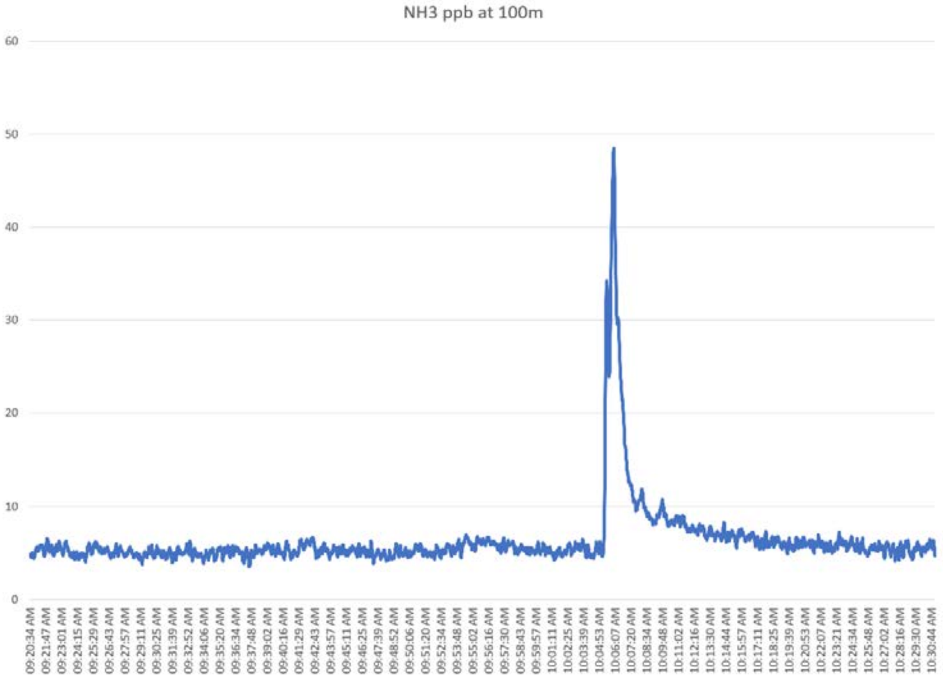


Figure 51. ICOS ammonia readings taken during the Wednesday morning camp fuel cook at 100 meters from the cook shed. The spike in ammonia concentration observed at 10:06 am corresponds to the bottle failure that occurred during this cook.

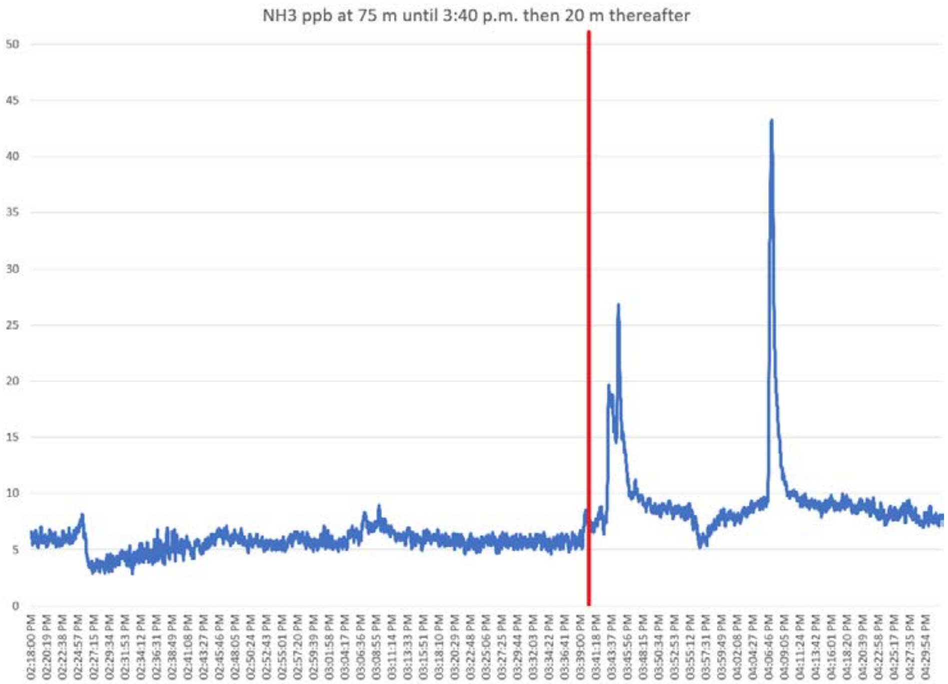


Figure 52. ICOS ammonia readings taken during the Wednesday afternoon ether cook at 75 meters from the cook shed. Red line marks ICOS relocation to 20 meters downwind of shed.

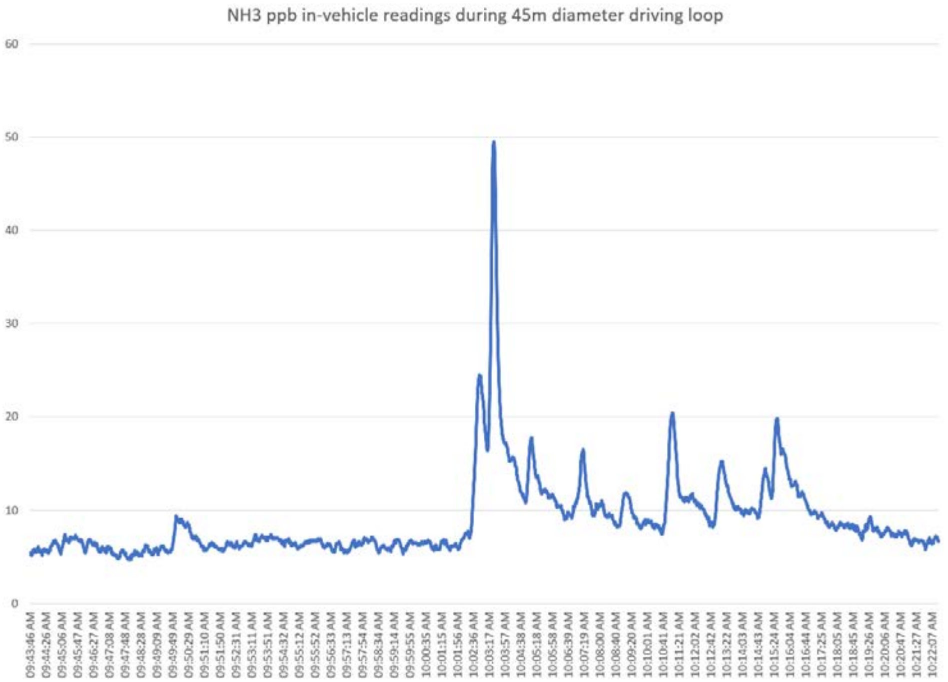


Figure 53. ICOS ammonia readings taken during the Thursday morning camp fuel cook from in-vehicle placement during continuously driven circuit of approximately 45 meters diameter and tangential to downwind side of shed. Ammonia spikes became pronounced after bottle failure and cook recovery to a food storage container that occurred shortly after 10 am. Peaks arose as the moving vehicle approached, and passed the live cook shed. Each circuit lap was approximately 2 minutes at low speed.

Sample[0] = Thu Nov 30 17 10:14:51.994

Sample[1000] = Thu Nov 30 17 09:58:28.132

Sample[0] = Thu Nov 30 17 10:14:51.994

Sample[1000] = Thu Nov 30 17 09:58:28.132

[H_2O] = 6998.16 PPM [NH_3] = 11.22 PPB [HCl] = 0.21 PPB

Time: Thu Nov 30 10:14:52 2017

Gas Temperature: 16.66 °C

Laser A τ: 20.69 µs

Figure 54. Bottom plot depicts ammonia peaks captured during 6 vehicle passes during the Thursday morning camp fuel cook. A new rising ammonia edge begins at far left during vehicle tangential approach to rear of cook shed (seen in top right) during this 7th driving pass as viewed on the 15-minute data screen.

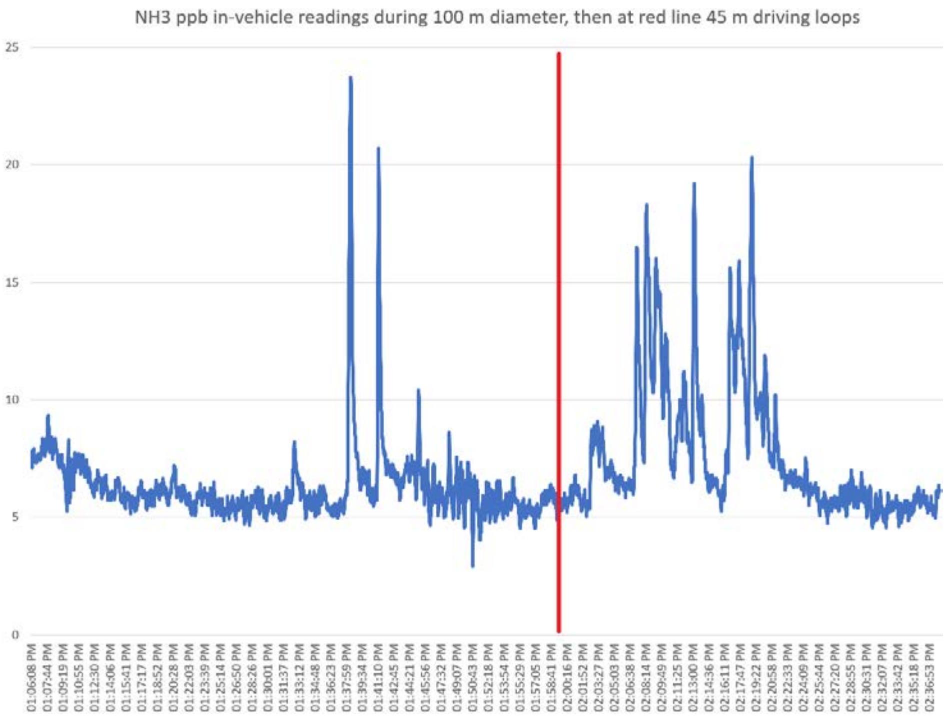


Figure 55. ICOS ammonia readings taken during the Thursday afternoon ether cook from in-vehicle placement during continuously driven circuit of first 100 meters diameter and tangential to downwind side of shed, then shortened at 1:59 pm (red line) to 45 meters diameter.

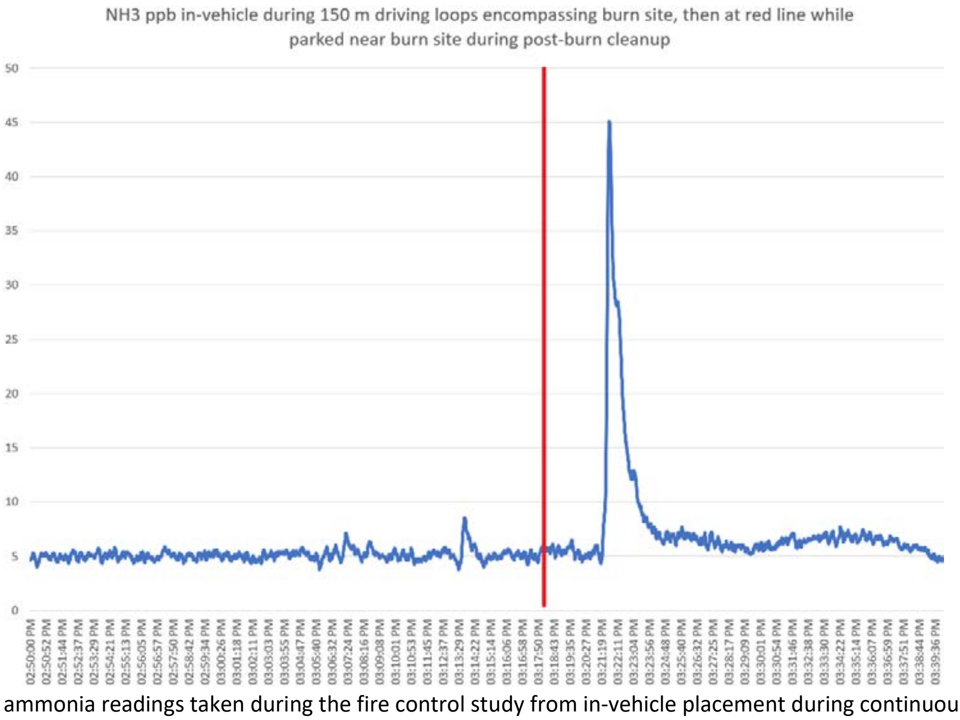


Figure 56. ICOS ammonia readings taken during the fire control study from in-vehicle placement during continuously driven circuit over 150 meters diameter, then parked near burn site at 3:18 pm (red line) following completion of burns and throughout cleanup.

Figure 57. Yellow cart containing battery-powered ICOS NH_3-HCl gas analyzer and external vacuum pump placed 40 meters downwind from cook shed during Tuesday afternoon camp fuel cook.

Table 12. ICOS distance from the cook shed during ammonia sampling.

Date	Cook type	Chronology	ICOS standoff	Notes
Nov 28 Tuesday PM	Camp Fuel	Cook start approx. 2:40 pm	40 m	Burp spikes evident
Nov 29 Wednesday AM	Ether	Cook start time approx. 9:25-9:41 am then bottle failure 10:04 am; fresh camp fuel cook from approximately 11:00 am – 12:30 pm	100 m	Bottle failure prominent spike
Nov 29 Wednesday PM	Ether	Cook start approx. 2:10 pm	75 m from cook start until 3:40 pm, then relocated to 20 m	ICOS switched on 1418. Wind shift predicated move to 20 m
Nov 30 Thursday AM	Camp Fuel	9:30 am cook begun - bottle failure Camp Fuel cook, salvaged to food storage container and continued	Moving vehicle	Driven loops tangential to downwind side of cook shed
Nov 30 Thursday PM	Ether	Cook start about 1:05 pm	Moving vehicle	
Nov 30 Thursday PM	Fire Control Study		Moving vehicle during burn; parked vehicle during post-burn cleanup	NH_3 signal greatest during cleanup steps

VOC Detection

The GMAP unit PID and the SPods were utilized for analysis of VOCs that were off-gassed from the One Pot methamphetamine cooks. All 4 SPods used in this study were uncalibrated so values should be treated as qualitative only. The 3 EPA-ORD SPods reported PID data as raw analog-to-digital converter counts (DAQ counts) while the SenSevere SPod reported the PID data in ppb as isobutylene. As the SPods are still in the research and development phase, there are still operational issues associated with the experimental nature of these new detectors. Data files collected by EPA's SPod C were corrupted, resulting in no data being present for this SPod.

For the PID installed on the GMAP unit, VOCs were identified between 14 and 2854 ppb isobutylene; the GMAP unit was parked at 15 m from the cook shed and 30 m from the fire control study. For the SPods, at 25 m the VOCs were identified between 411 and 6030 DAQ counts, at 50 m the VOCs were identified between 126 and 2697 DAQ counts, at 75 m the VOCs were identified between 117 and 594 DAQ counts, and at 100 m the VOCs were identified between 57.5 and 67.6 ppb isobutylene. Table 13 shows the average, minimum, and maximum VOC concentrations measured by the GMAP and SPod PIDs during each One Pot methamphetamine cook. In general, a higher concentration of VOCs were observed during the One Pots that used camp fuel as an organic solvent. This is to be expected, as PIDs generally have a better response to long saturated hydrocarbons than to short saturated hydrocarbons.[16] Camp fuel is a mixture of many saturated hydrocarbons, most of which have molecular structures that range from 5 to over 11 carbons in length.[17] The starting fluid used for the ether One Pot methamphetamine cooks is composed of 40-70 percent ethyl ether and 25-60 percent heptane.[18] While the ethyl ether and heptane can be observed with a PID, they will generate a lower detector response that the long, saturated hydrocarbons from the camp fuel.

Figures 58-79 show the VOC concentrations measured by the GMAP unit and SPod PIDs during each One Pot event. Overall, more spikes in VOC concentration were observed during the ether cooks. The larger number of spikes is likely due to the greater volatility of ether when compared to camp fuel, allowing more ether to be released from the One Pot during burping. While more spikes in the VOC concentration were observed in the ether cooks, the camp fuel cooks had a larger concentration of VOCs present when a spike was observed, which was usually during filtration of the One Pot.

Table 13. The average, minimum, and maximum VOC concentrations measured by the GMAP unit and SPod PIDs during each One Pot methamphetamine cook.

		Sampling Event							
		Ether 1	**Camp Fuel 1**	**Camp Fuel Fail 1**	**Camp Fuel 2**	**Ether 2**	**Camp Fuel Fail 2**	**Ether 3**	**Fire Control Study**
GMAP VOC Concentration (ppb Isobutylene)	**Average**	378	703	504	304	245	229	70	26
	Maximum	991	1850	2041	402	468	2854	284	81
	Minimum	278	522	373	267	216	115	35	14
25 m SPod VOC Concentration (DAQ Counts)	**Average**	973	1058	1005	798	546	-	-	-
	Maximum	2430	5737	6030	2131	1054	-	-	-
	Minimum	905	884	804	743	411	-	-	-
50 m SPod VOC Concentration (DAQ Counts)	**Average**	737	691	400	162	137	730	926	932
	Maximum	1072	2697	2134	200	153	2041	1076	946
	Minimum	682	638	280	133	126	624	897	912
75 m SPod VOC Concentration (DAQ Counts)	**Average**	-	-	-	-	-	137	461	573
	Maximum	-	-	-	-	-	162	594	594
	Minimum	-	-	-	-	-	117	374	548
100 m SPod VOC Concentration (ppb Isobutylene)	**Average**	64.4	64.6	60.7	57.9	58.5	63.1	63.6	63.5
	Maximum	65.5	67.6	65.8	58.6	59.0	65.2	64.3	64.0
	Minimum	63.8	64.1	59.6	57.5	58.0	60.4	63.0	63.1

Figures 58-60 show the VOC concentrations measured by the GMAP unit and SPod PIDs during the first ether One Pot methamphetamine cook. The high VOC concentration observed at the beginning of the cook with the GMAP and SenSevere SPod is due to the loss of starting fluid during the depressurization of the cans (Figures 58-60). This increase can also be observed with the EPA-ORD SPod, but at a lesser extent (Figure 59). The spike in VOCs observed near 10:25 correspond to the start of the first ether cook and the spike near 11:45 correspond to filtration of the One Pot, just prior to salting out the methamphetamine. During both of these times, solvent is sitting in containers that are open to the environment, thus allowing more VOCs to be dispersed into the air than is observed during the rest of the cook. The spikes in VOC concentration during salting out is not due to the presence of HCl gas, as HCl does not readily ionize in PIDs so the instrument cannot detect it.[16] The small spikes observed around 10:35 and 10:50 correspond to times when sodium hydroxide was added to the One Pot. During these events, the cap was removed from the One Pot so sodium hydroxide could be added to the reaction, allowing any VOCs that were in the headspace of the reaction to escape into the environment. As can be seen in Figure 60, plumes of VOCs could be detected as far as 100 m downwind of the One Pot, though the burping events could not be detected at this distance.

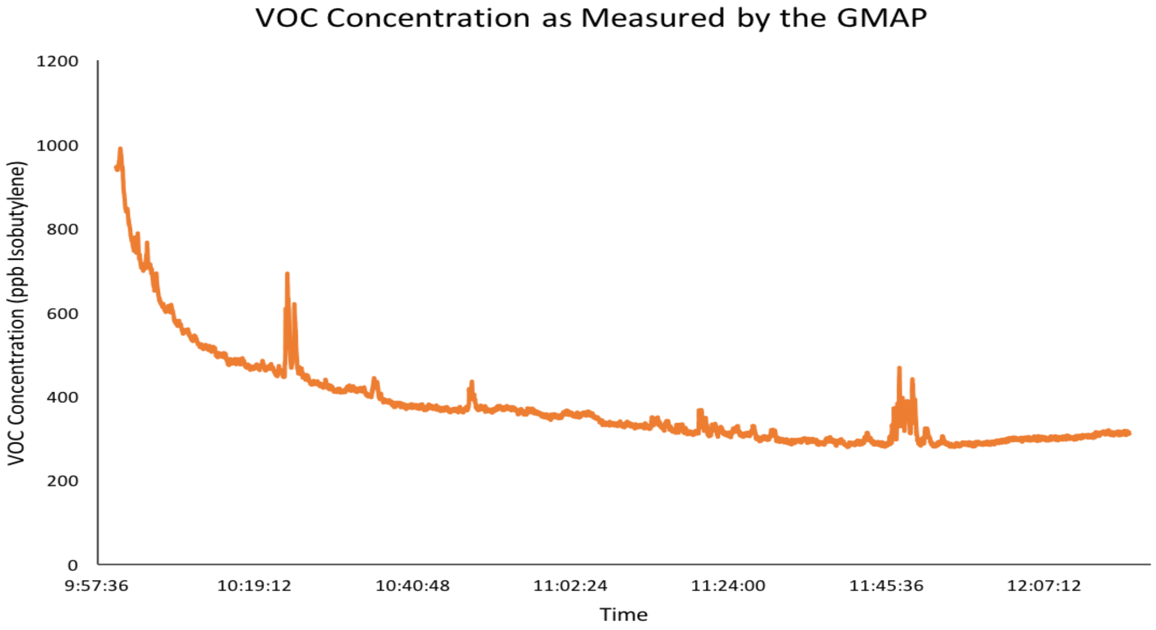


Figure 58. VOC concentrations measured by the GMAP unit PID during the first ether One Pot methamphetamine cook.

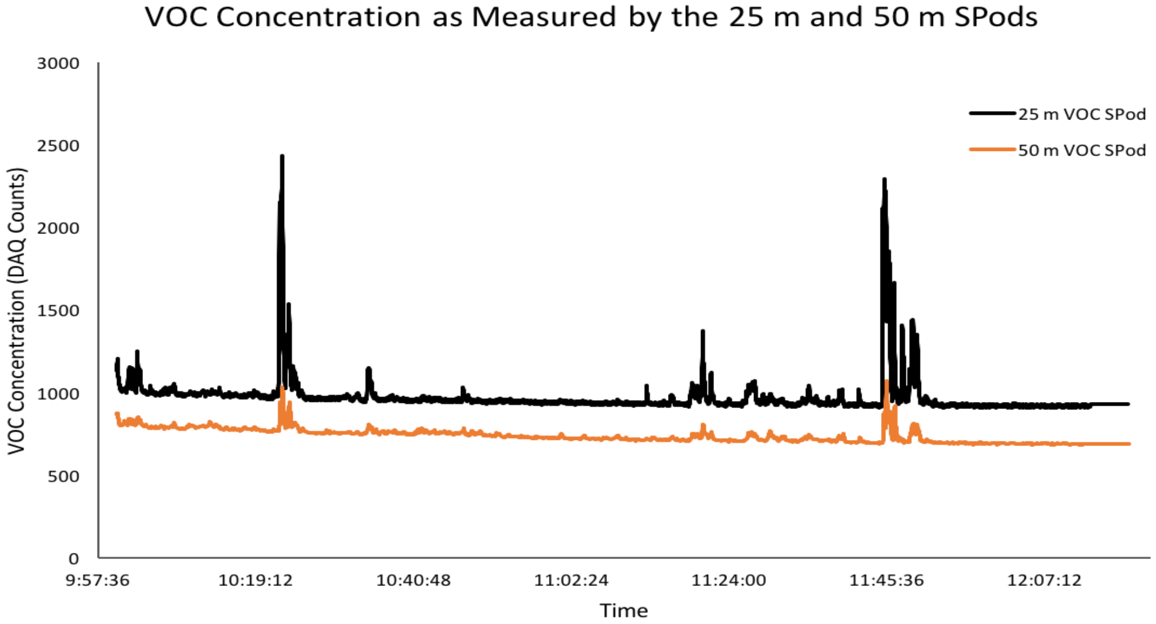


Figure 59. VOC concentrations measured by the EPA-ORD SPods during the first ether One Pot methamphetamine cook.

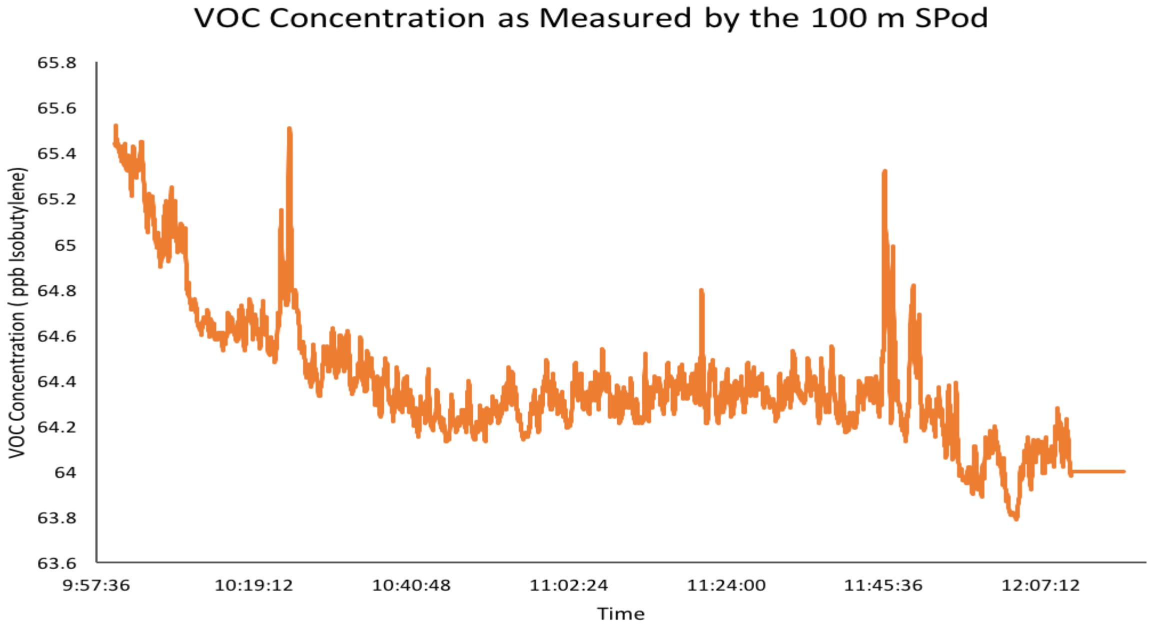


Figure 60. VOC concentrations measured by the SenSevere SPod during the first ether One Pot methamphetamine cook.

Figures 61-63 show the VOC concentrations measured by the GMAP unit and SPod PIDs during the first camp fuel One Pot methamphetamine cook. The concentration of VOCs present in the atmosphere begin increasing around 16:00, which is when the One Pot was filtered and salting out began. No other spikes in VOC concentration were observed during this One Pot, however, the baseline level of VOCs in the atmosphere was elevated slightly when compared to the ether One Pot performed earlier that morning. The absence of spikes in VOC concentration during burping events throughout the cook is attributed to the volatility of camp fuel when compared to ether. Ether is more volatile than camp fuel, causing more of this solvent to be released during burping than when camp fuel is used. The increased baseline level of VOCs is likely due to a decrease in wind speed observed during the afternoon of the camp fuel cook. The gentler breeze present during the camp fuel One Pot would cause the air, and thus the VOCs, to move less and would allow more of the VOCs to interact with the PID detector, resulting in a slightly elevated baseline level of VOCs.

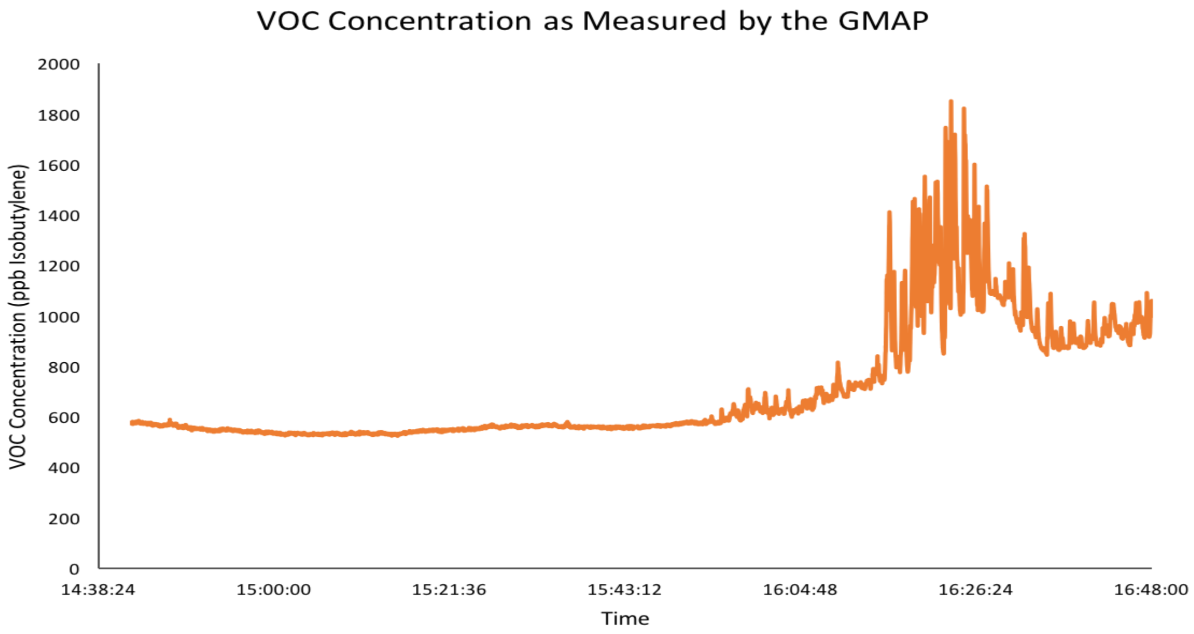


Figure 61. VOC concentrations measured by the GMAP unit PID during the first camp fuel One Pot methamphetamine cook.

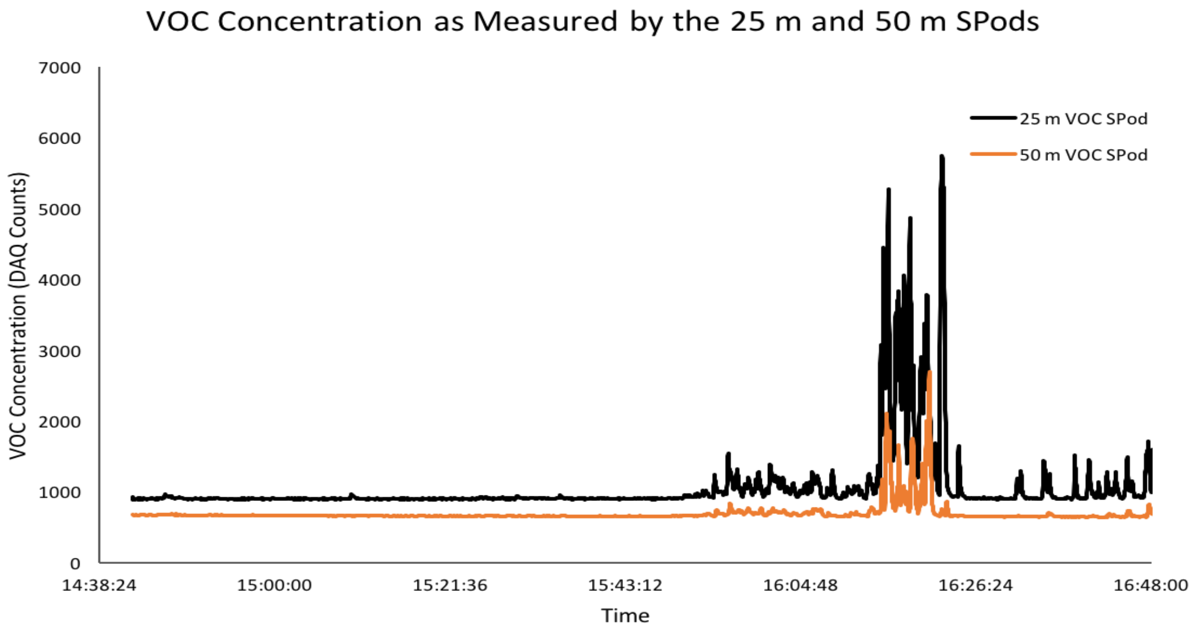


Figure 62. VOC concentrations measured by the SPods during the first camp fuel One Pot methamphetamine cook.

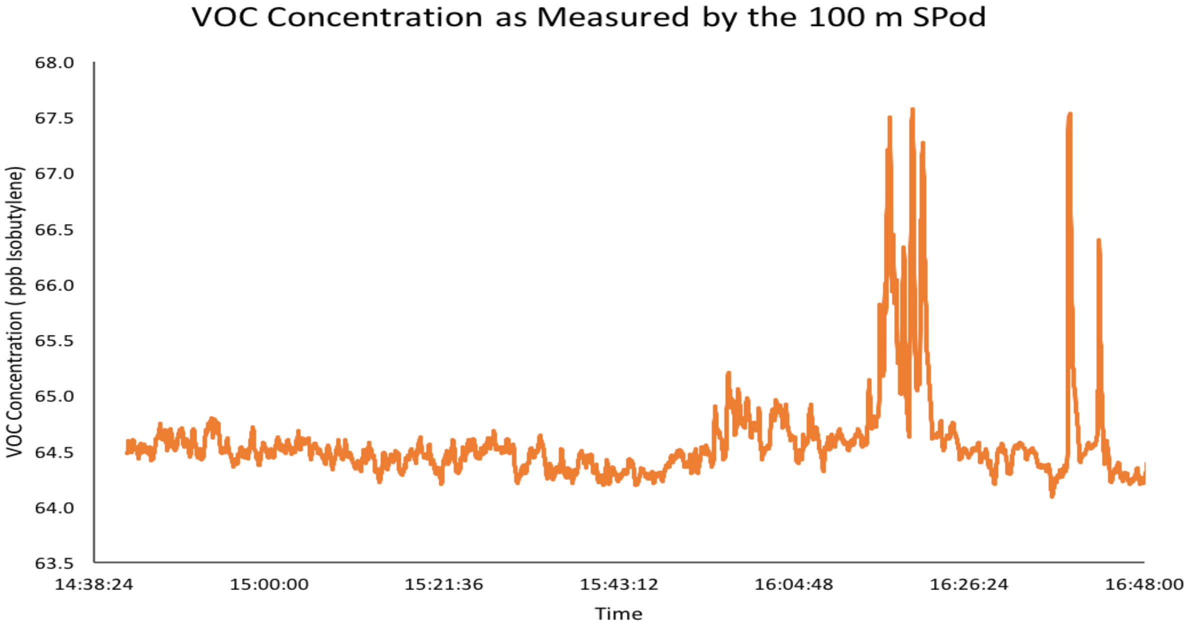


Figure 63. VOC concentrations measured by the SenSevere SPod during the first camp fuel One Pot methamphetamine cook.

Figures 64-66 show the VOC concentrations measured by the GMAP unit and Spod PIDs during the first One Pot bottle failure. The large spike in VOC concentration around 10:10 correlates with when the structural integrity of the bottle was compromised, causing camp fuel to be sprayed into the air.

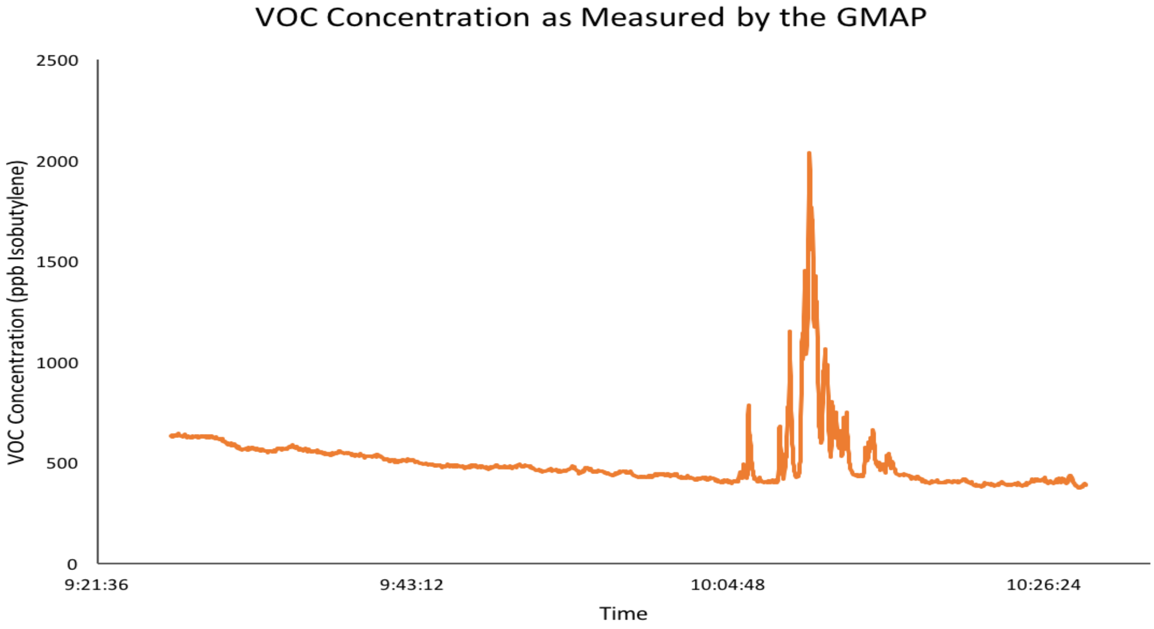


Figure 64. VOC concentrations measured by the GMAP unit PID during the first bottle failure.

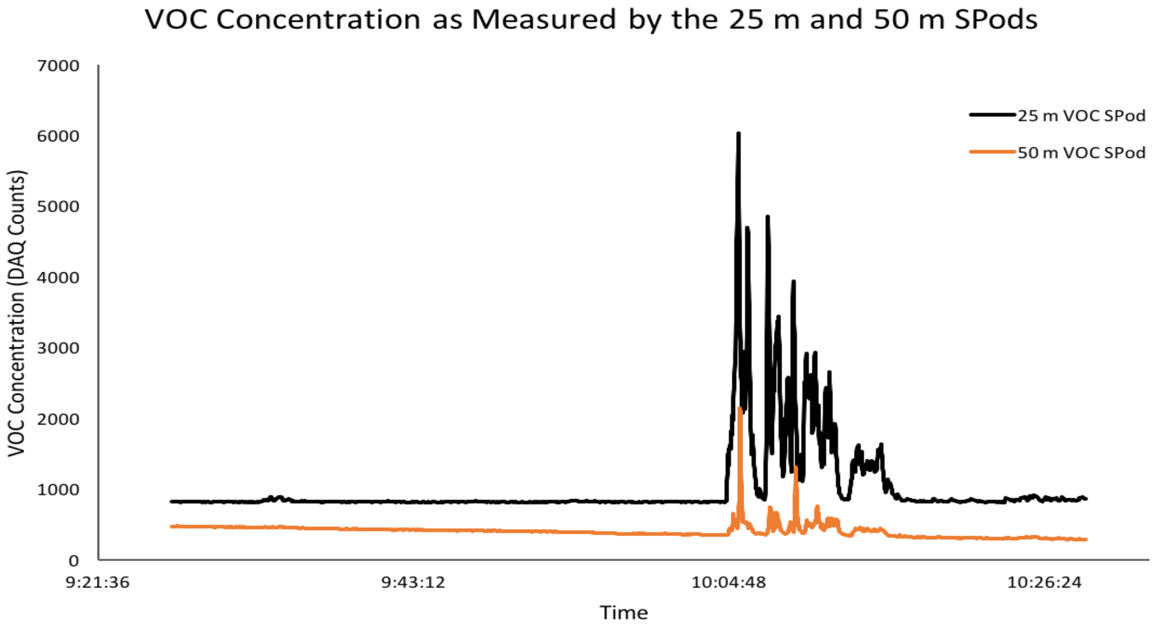


Figure 65. VOC concentrations measured by the SPods during the first bottle failure.

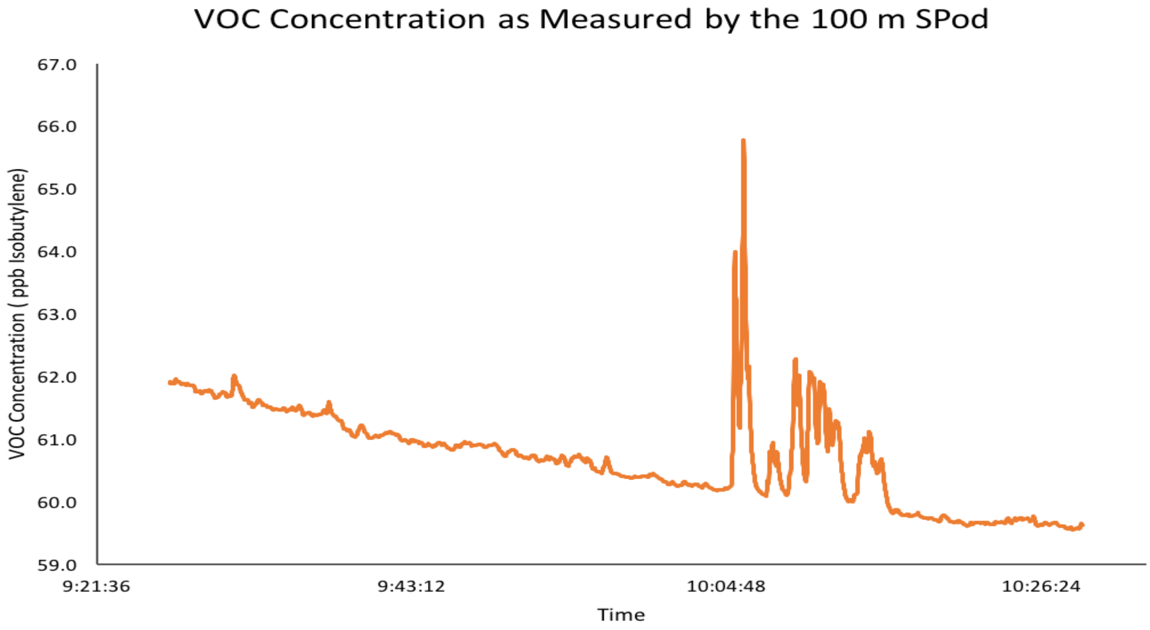


Figure 66. VOC concentrations measured by the SenSevere SPod during the first bottle failure.

Following the bottle failure, another camp fuel One Pot methamphetamine cook was immediately started. The VOC concentrations measured by the GMAP unit and SPod PIDs during this second camp fuel cook can be seen in Figures 67-69. The GMAP unit PID observed no significant spike in the VOC concentration during the second camp fuel cook (Figure 67), but the 25 m EPA-ORD SPod PID observed a large spike in VOC concentrations around 12:25 (Figure 68) and the 100 m SenSevere SPod PID observed a small spike in VOC concentration at the same time point (Figure 69). Prior to the second camp fuel cook, the GMAP unit was moved to better position it downwind of the One Pot cook shed, however the wind direction changed before the second One Pot began, but the GMAP was not moved again. Wind shift is therefore the most likely explanation for the GMAP PID not detecting VOCs emitted by the second camp fuel One Pot, but the EPA-ORD SPod at 25 m was still able to detect an increased concentration of VOCs during the filtration of the One Pot. The decrease in VOC concentration observed by the SenSevere SPod over the first 45 minutes of the second camp fuel One Pot is likely due to a raised background level of VOCs from the bottle failure that occurred immediately prior to the successful camp fuel cook. The SenSevere SPod was positioned far enough from the cook shed that it was able to detect smaller changes in VOCs than the SPods positioned close to the cook shed, which had larger concentrations of VOCs in the surrounding environment, masking small changes in the VOC concentration.

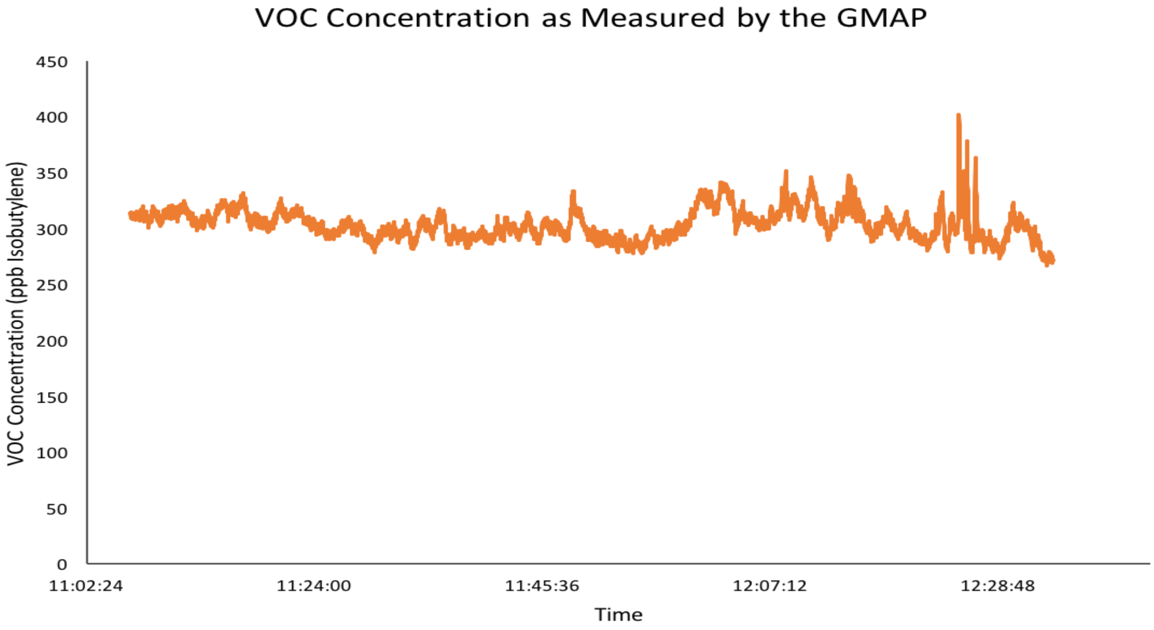


Figure 67. VOC concentrations measured by the GMAP unit PID during the second camp fuel One Pot methamphetamine cook.

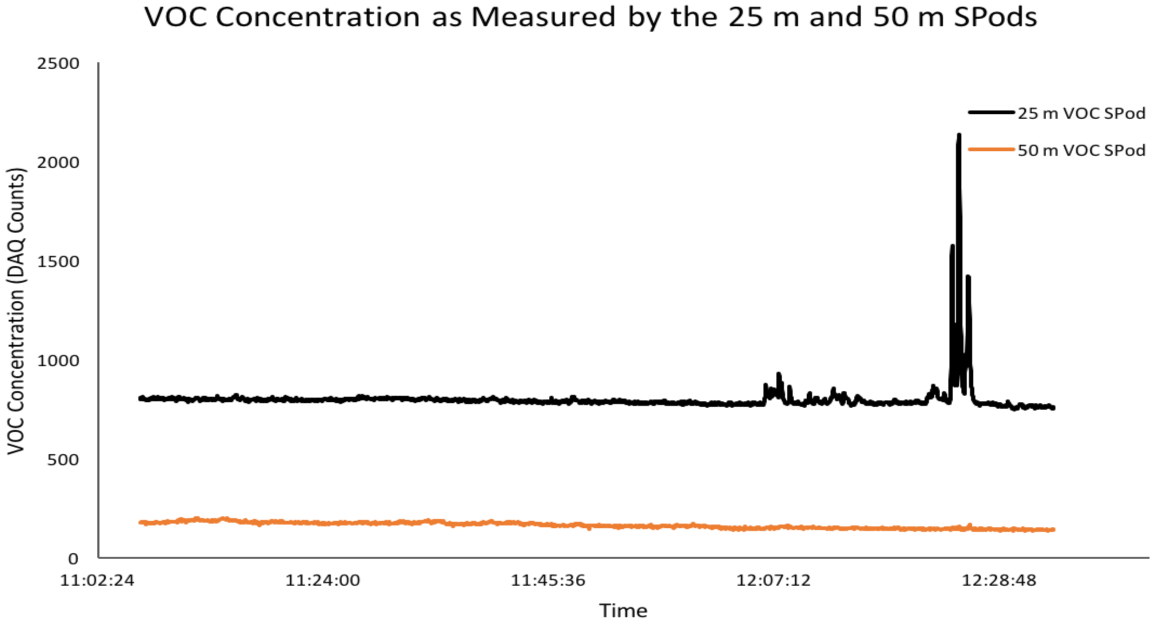


Figure 68. VOC concentrations measured by the SPods during the second camp fuel One Pot methamphetamine cook.

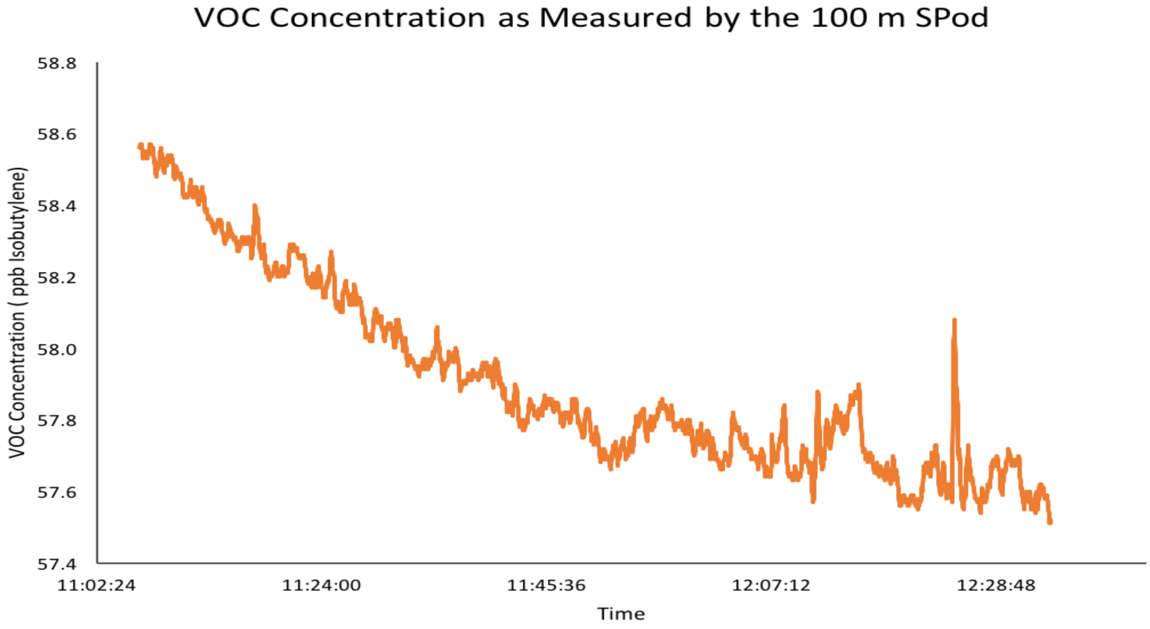


Figure 69. VOC concentrations measured by the SenSevere SPod during the second camp fuel One Pot methamphetamine cook.

Figures 70-72 show the VOC concentrations measured by the GMAP unit and SPod PIDs during the second ether cook. The PIDs were activated after the cans of starting fluid were depressurized, so the immediate increase in VOC concentration observed in the first ether cook was not observed by the GMAP PID (Figure 70), but this spike in VOC concentration at approximately 14:25 was observed as it diffused out to the 25 m EPA-ORD SPod (Figure 71). Due to the low level of VOCs present in the air during the second ether cook, small spikes in VOC concentration can be observed with the GMAP PID and the 25 m EPA-ORD SPod at several time points, such as around 15:05 and 15:21. These small spikes in VOC concentration correspond to times when the One Pot methamphetamine lab was burped, which was at 5 minute intervals. The large spike in VOC concentration observed around 15:40 was when the One Pot was filtered and the organic solvent was in a container open to the atmosphere. For the second ether One Pot methamphetamine cook, no significant spike in VOC concentration was observed with the SenSevere SPod located 100 m from the cook shed (Figure 72).

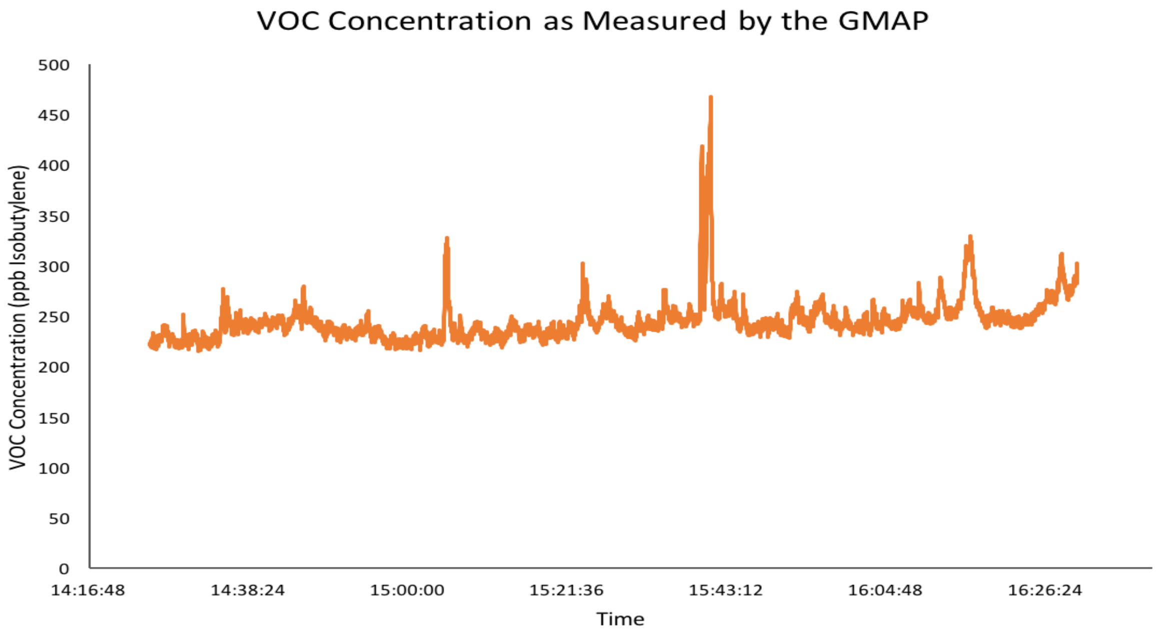


Figure 70. VOC concentrations measured by the GMAP unit PID during the second ether One Pot methamphetamine cook.

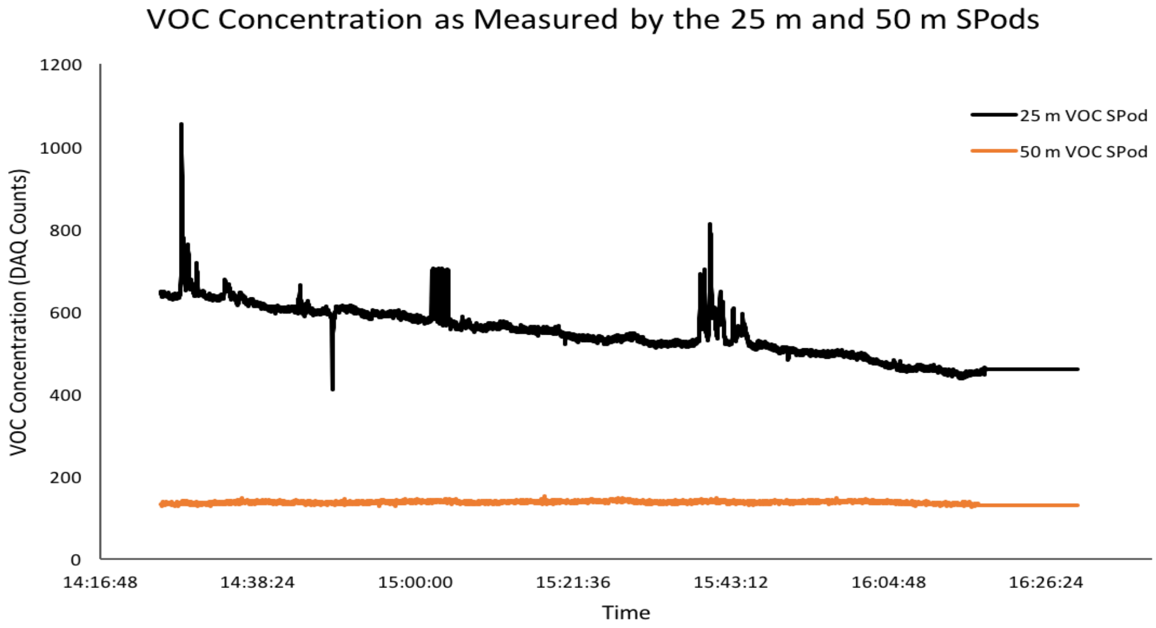


Figure 71. VOC concentrations measured by the SPods during the second ether One Pot methamphetamine cook.

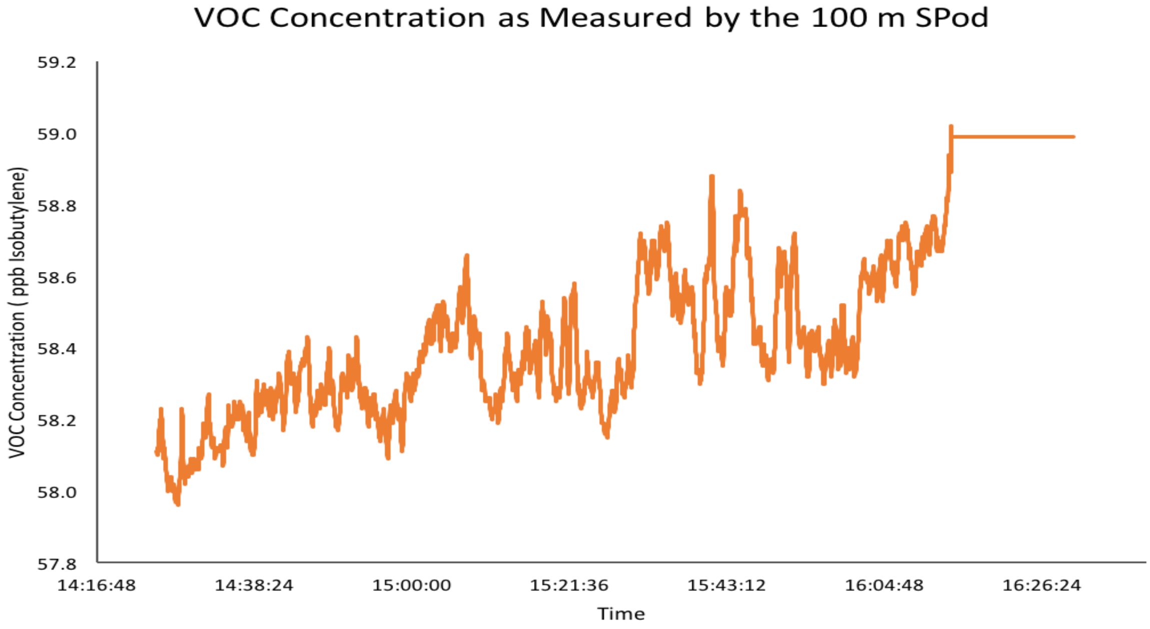


Figure 72. VOC concentrations measured by the SenSevere SPod during the second ether One Pot methamphetamine cook.

Figures 73-75 show the VOC concentrations measured by the GMAP unit and SPod PIDs during the second One Pot bottle failure during the Thursday morning camp fuel cook. As with the first bottle failure, a large spike in VOC concentration is observed at the time point in which the structural integrity of the bottle was compromised. This is seen around 10:00. Unlike the first bottle failure, after the second bottle failure, the contents of the One Pot methamphetamine lab were dumped into a food storage container, along with another instant cold pack and additional Drain Out, and the methamphetamine cook was allowed to continue. A second spike in VOC concentration is observed around 11:00, which is when this modified One Pot was filtered and the organic solvent was left open to the environment.

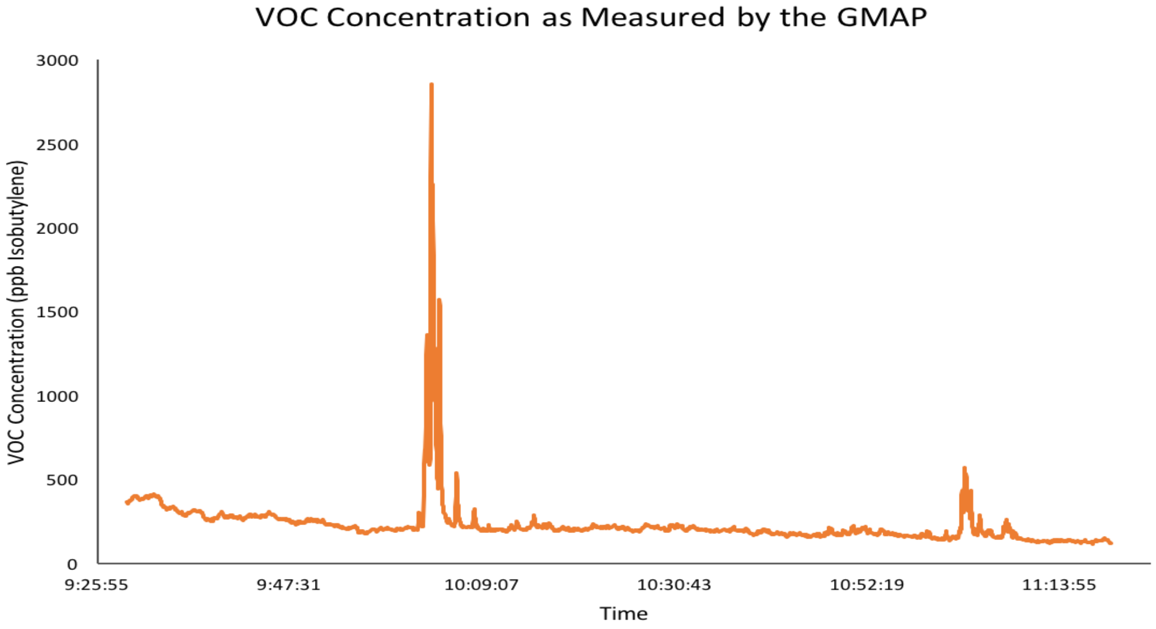


Figure 73. VOC concentrations measured by the GMAP unit PID during the camp fuel cook, including a second bottle failure.

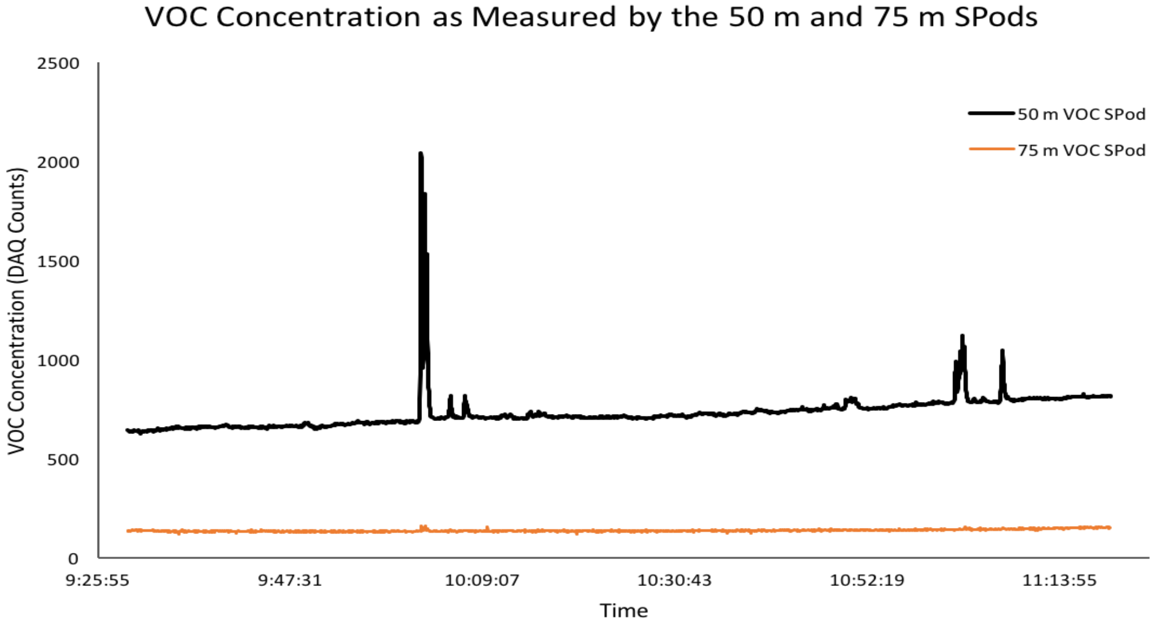


Figure 74. VOC concentrations measured by the SPods during Thursday morning's camp fuel cook, including a second bottle failure.

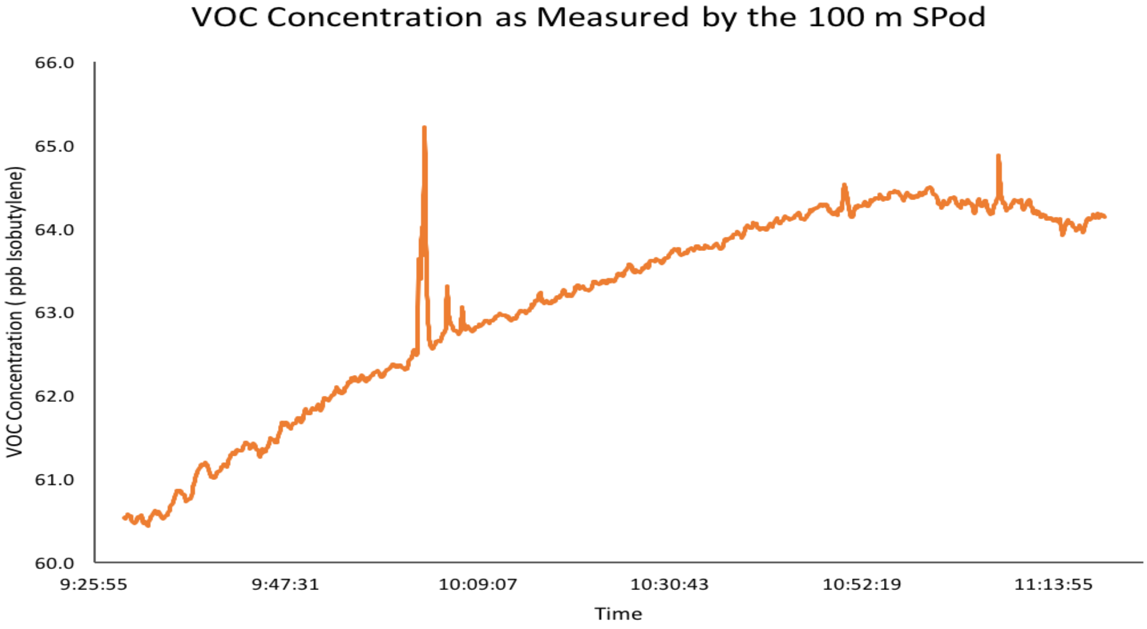


Figure 75. VOC concentrations measured by the SenSevere SPod during Thursday morning's camp fuel cook, including a second bottle failure.

Figures 76-78 show the VOC concentrations measured by the GMAP unit and SPod PIDs during the third ether One Pot methamphetamine cook. As with the second ether cook, the GMAP PID was not activated until the starting fluid cans had been depressurized, thus missing the large, initial VOC spike. Also, as with the second ether cook, the background VOC concentrations observed during the third ether cook were relatively low, enabling the spikes in VOC concentration corresponding to the burping of the One Pot to be observed by the GMAP unit PID (Figure 76) and the EPA-ORD SPods located at 50 m; the 75 m SPod (Figure 77) appears to have experienced instrument drift during this cook but indicates a small perturbation at 13:40 that is likely due to a One Pot burp. The spike in VOC concentration at 13:40 observed with the SenSevere SPod located 100 m from the cook shed (Figure 78) is also correlated to the burp event seen across all 4 PIDs. The large spikes in VOC concentration observed around 14:12 are from the filtration of the One Pot and the organic solvent being opened to the environment.

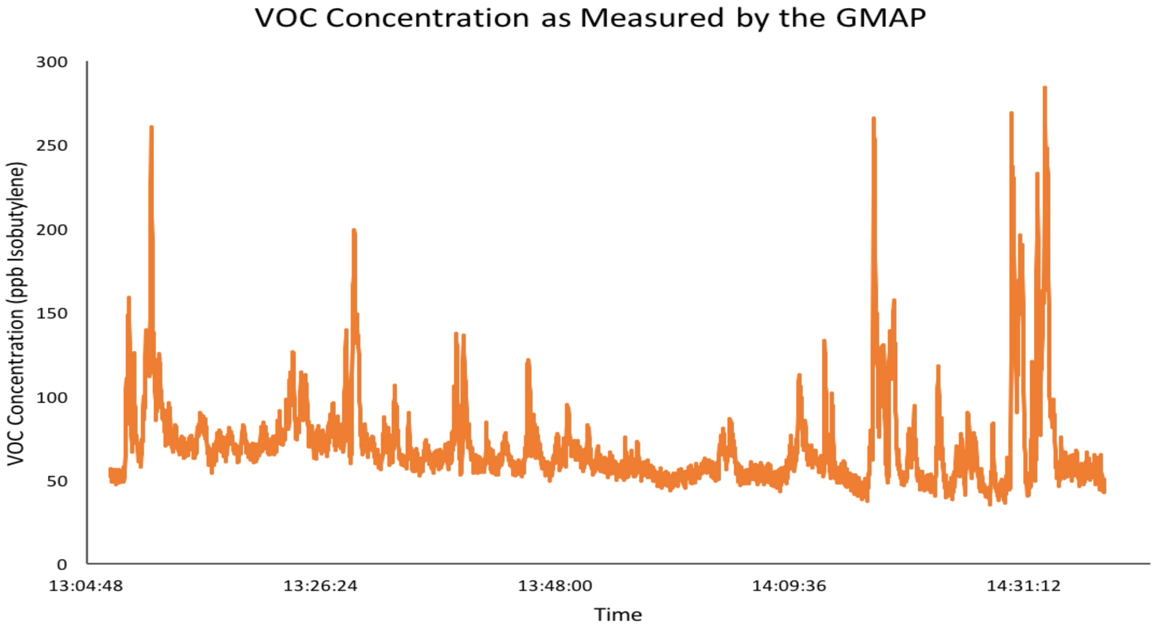


Figure 76. VOC concentrations measured by the GMAP unit PID during the third ether One Pot methamphetamine cook.

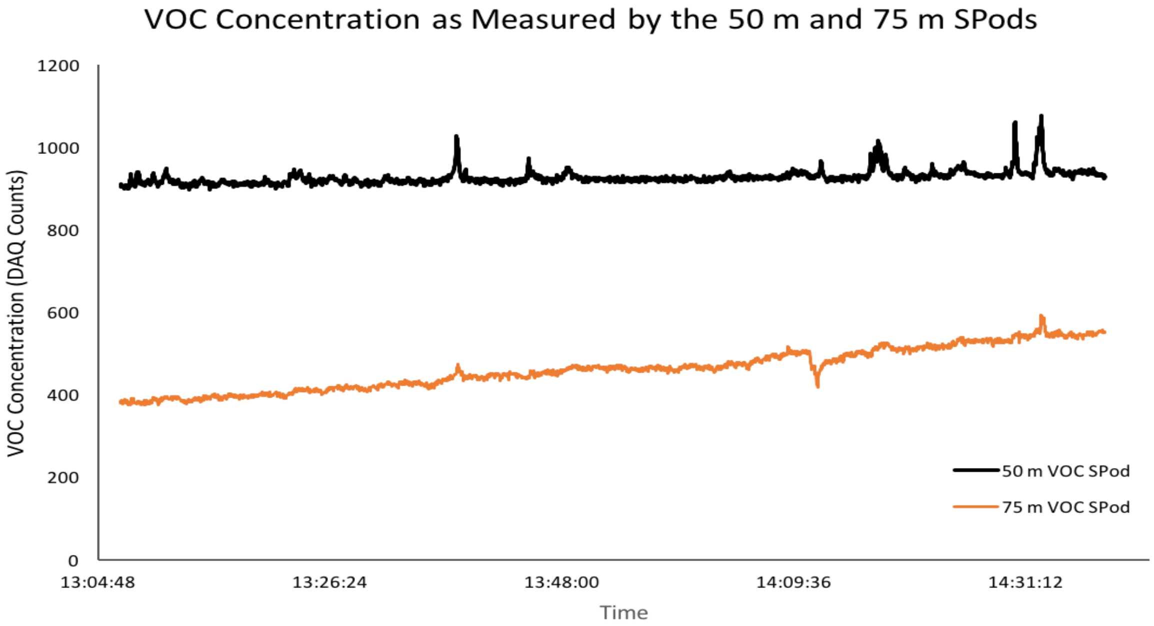


Figure 77. VOC concentrations measured by the SPods during the third ether One Pot methamphetamine cook.

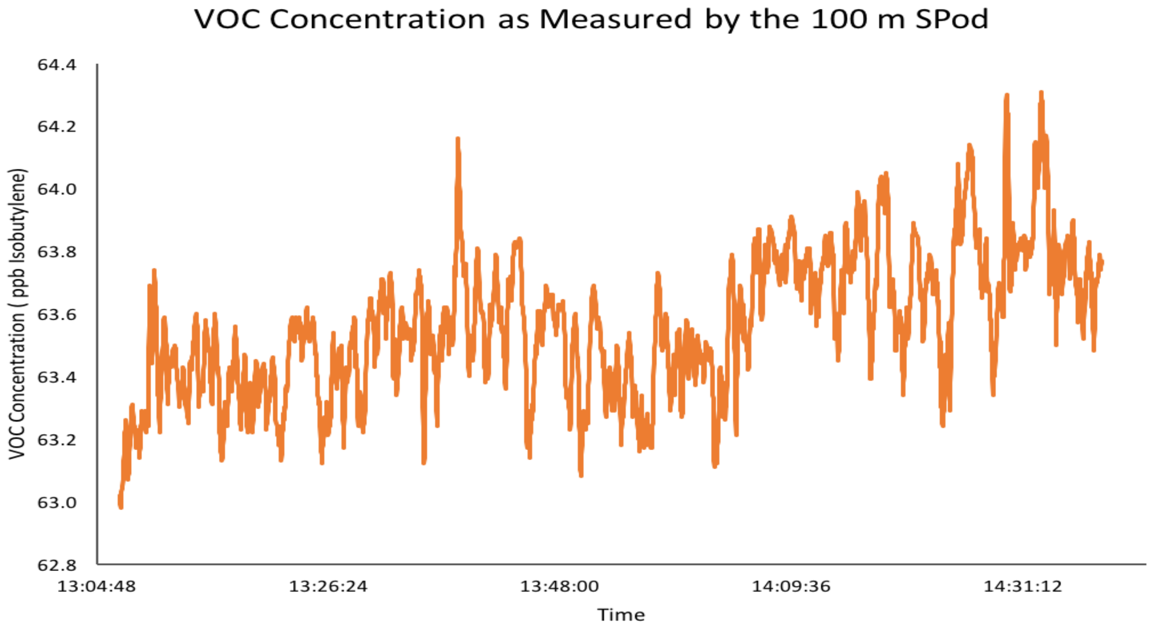


Figure 78. VOC concentrations measured by the SenSevere SPod during the third ether One Pot methamphetamine cook.

Figure 79 shows the VOC concentrations measured by the GMAP unit PID during the One Pot fire control study. Very little variation in the VOC concentration was observed in this study excepts for a single, large spike around 14:46. This single spike corresponds to the first ether One Pot that was set on fire and allowed to burn to completion to give the researchers an idea of what a typical One Pot fire may look like. As can be seen in Figure 80, during this fire, the bottle broke open and solvent formed a burning puddle around the One Pot. This leakage of solvent is correlated in time to the spike in VOC concentration in Figure 79. For the remaining One Pot burns, the fire was doused with one of the firefighting techniques before the bottle had significant solvent leakage. This no doubt prevented a sudden plume of VOCs from being released into the atmosphere and likely explains the negligible variations in VOC concentration observed during the One Pot fire control study.

While data was collected by the SPods during the fire control experiment, the SPods were not moved following the third ether One Pot methamphetamine cook. By not moving the SPods, the closest SPod was 85 m from the burn site while the other two working SPods were 110 and 135 m from the burn site. During the fire control tests, no spikes in VOC concentration were observed by any of the SPods.

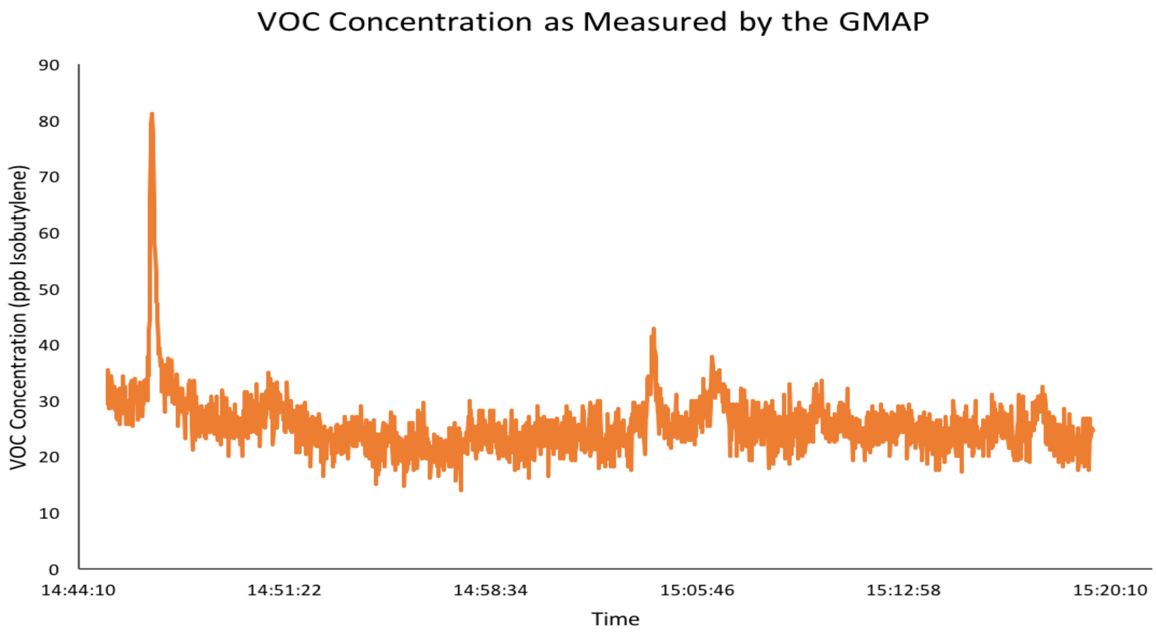


Figure 79. VOC concentrations measured by the GMAP unit PID during the One Pot fire control study.

Figure 80. The first One Pot burn during the fire control study. As can be seen, the bottle broke open during the fire, causing a puddle of ether to surround the burning bottle and ignite.

To supplement the VOC data collected with the GMAP unit and SPod PIDs, a forward looking infrared (FLIR) video camera was used to visualize the VOCs released during production of the One Pot methamphetamine labs. Figure 81 shows still photographs taken from a video captured by the FLIR camera as two of the researchers hold and burp a One Pot. The first photograph was taken right as the One Pot was burped and a small cloud of VOCs can be observed beginning to surround the One Pot. In the second photograph, taken one second after initiation of the burp, the cloud of VOCs can be seen surrounding the researcher's hands and forearms. In the third paragraph, the cloud of VOCs can be seen engulfing the researchers and is filling the headspace of the cook shed. Other videos from the FLIR camera allowed the researchers to visualize VOCs seen escaping the shed through cracks in the floor and walls, as well as evident visualization of rapid VOCs diffusion once they escaped the shed and were mixed into the atmosphere by the wind.

Figure 81. Still shots taken by the FLIR camera during a One Pot burp. a.) a One Pot as it is burped. b.) a One Pot one second after it was burped. c.) a One Pot six seconds after it was burped.

HCl Detection

The portable ICOS NH_3-HCl gas analyzer and data-logger responded poorly to the HCl generated during the salting out of the methamphetamine. To test the instrument's response to HCl gas, a large plume of HCl gas was generated by combining bulk sodium chloride and concentrated sulfuric acid in an open 5 gallon bucket. The ICOS was placed 5 m downwind of the bucket containing the source of HCl gas, but the instrument only registered a low ppb level, as shown in Figure 82.

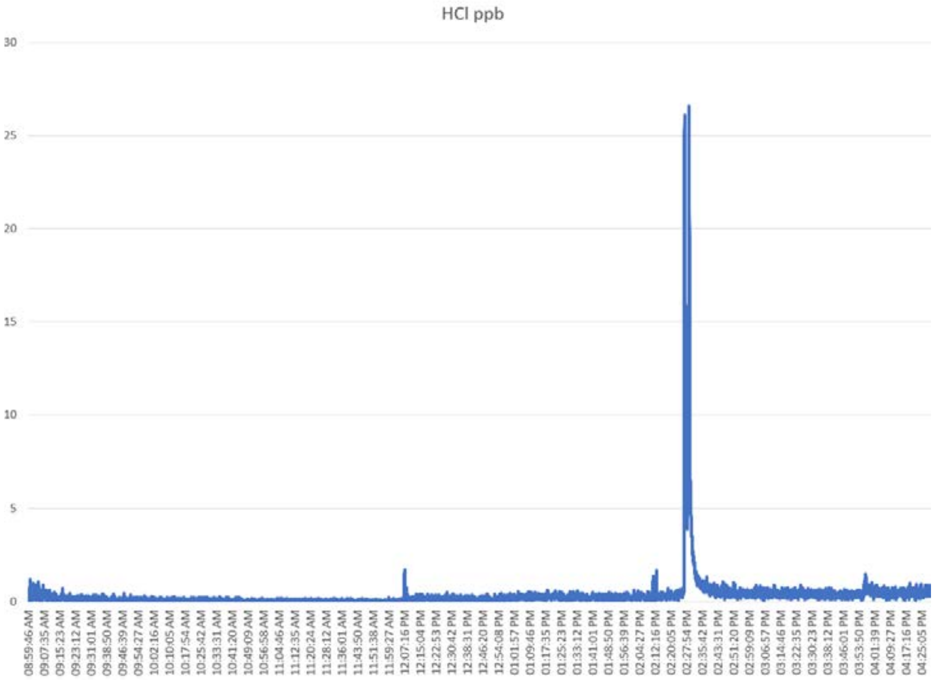


Figure 82. Poor HCl response during salting out resulted in an experimental open-bucket reaction of sodium chloride with concentrated sulfuric acid, seen as the double peak around 2:30 pm Wednesday. A far larger hydrogen chloride concentration was likely to be present at the gas analyzer inlet than the low ppb response seen here.

Fire Control Study

For the One Pot fire control study, effectiveness of a firefighting agent was assessed by its ability to contain a fire and keep it from spreading, as well as its ability to extinguish the fire. These assessments were qualitative only, as the assessments were made from visual observations by the researchers who were present during the burns. For comparison, two One Pots were set on fire at the beginning of the study, one with camp fuel as a solvent and one with ether as a solvent. This allowed for a better perspective on what each One Pot methamphetamine lab fire might look like when assessing the effectiveness of the three firefighting agents tested during this study.

The first firefighting technique examined in this study was Cold Fire^{TM} Tactical, which is marketed to law enforcement as an easy to use, personal fire extinguisher fit to put out types A, B, and D fires by removing the heat and fuel source from the fire itself.[19] During this study, the researchers observed that Cold Fire^{TM} was fairly effective in containing the fire so that only the One Pot itself was burning, but was ultimately ineffective at extinguishing the fire. The whole can of Cold Fire^{TM} Tacticle was used during this study in an attempt to extinguish the fire. This product may be best suited as a means to contain a fire at a One Pot methamphetamine lab while first responders evacuate the building and wait for firefighters to arrive on the scene as opposed to being used as a means to fully extinguish a One Pot methamphetamine lab fire.

The second fire study technique examined in this study was use of Imbiber Beads®, which are marketed as a super absorbent polymer that can absorb up to 27 times its volume in organic solvent.[20] These beads are currently suggested by the DEA as the best method for stabilizing One Pot methamphetamine labs and minimizing their fire risks.[21] In our study, the Beads effectively absorbed the flammable waste solvent, but once this stabilized waste was externally lit, the Imbiber Beads® were found to be readily flammable. When the Imbiber Beads® were poured into the spent One Pots, they absorbed the organic solvent from the One Pot and became a gelatinous substance. Even with the organic solvent in this gelatinous state, the One Pots were still easily ignited and the resulting fire burned for some time after ignition. The Imbiber Beads® may aid in fire prevention by reducing the volume of spillable solvent and enveloping the air-flammable lithium from the One Pot within the gelatinous organic solvent-Imbiber Bead® mixture, but we observed the absorbed solvent-bead mix is flammable when externally lit.

The final firefighting technique examined in this study was the use of a training fire extinguisher. A training fire extinguisher is comparable to a standard, class A dry powder fire extinguisher, but instead of the extinguisher being loaded with monoammonium phosphate, the training extinguisher is loaded with sodium bicarbonate. This type of fire extinguisher is less efficient at extinguishing fires than a standard dry powder extinguisher, but it is cheaper and better suited for training purposes. This study found that the training fire extinguisher excelled at both containing the One Pot methamphetamine lab fires, as well as extinguishing them. Based on what was observed during this fire control study, a small dry powder extinguisher may be an effective tool for first responders to extinguish a One Pot methamphetamine lab fire.

Conclusion

This study provided an in-depth look at contamination types and levels observed on-scene at a controlled, field-constructed One Pot methamphetamine laboratory and attempted live cook plume detection using fixed and mobile instruments. Seven One Pot methamphetamine cooks were performed at the Oklahoma State University Fire Research and Training Center in a plastic garden shed to simulate a residential cook location. Within the shed, contamination monitoring was provided through body-worn sample collectors and surface area swipe sampling. Air monitoring was set up inside and downwind of the shed to collect and identify the plume effluents from the One Pot methamphetamine cooks using ether and camp fuel as cook solvents. Air effluents identified during One Pot methamphetamine production include, but are not limited to, ammonia and numerous VOCs, including straight-chain hydrocarbons, aromatic and substituted aromatic hydrocarbons, ethyl ether, and methamphetamine. Effluent plumes were visualized using a FLIR camera as they were emitted during the One Pot cook, notably recording effluent clouds quickly engulfing the researchers and filling the cook shed. Ammonia plumes were detected at standoff distances using DUVAS, PID and ICOS field portable instruments, to include detection from stationary and moving vehicles.

Shed interior surface contamination was examined via swab sampling techniques before and after each cook and following a wet decontamination procedure. Lateral flow immunoassays were used as presumptive field tests for the presence of methamphetamine contamination pre-cook, post-cook and after a wet decontamination process was performed on the cook shed and the researcher's personal protective equipment. Samples derived from surface swipes were split and tested on-scene with the lateral flow immunoassays and the remainder was shipped to the CDC-NIOSH Taft laboratory in Cincinnati, OH for quantitative analysis of methamphetamine using a fluorescence covalent microbead immunosorbent assay. Quantitative analysis of the surface swabs found the wet decontamination process comprised resulted in no significant decrease in the amount of methamphetamine present on the PPE and shed surfaces.

At the end of this study, the One Pots were burned to investigate several popular products currently employed to combat or prevent One Pot methamphetamine lab fires. The techniques examined included use of a Cold Fire™ Tactical personal fire extinguisher, use of a training dry-powder fire extinguisher, and pre-fire use of Imbiber Beads® designed for flammable solvent waste absorption. It was found that the Cold Fire™ Tactical personal fire extinguisher was able to prevent the fire from spreading, but it was rather ineffective at fully extinguishing the fire. The training dry-powder fire extinguisher, while not as effective as a standard issue dry-powder extinguisher, quickly extinguished the One Pot methamphetamine lab fires and did not cause the fire to spread as it was being extinguished. From the observations made by this research group, it is suggested that standard fire extinguishers be employed to combat One Pot methamphetamine lab fires. The Imbiber Beads® were found to absorb the organic solvent from the One Pot and form a gelatinous substance. This appeared to prevent One Pot flashover fires, however, the bead mixture still readily burned, even in the presence of excess Imbiber Beads®.

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