JAY INSLEE
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Chief

STATE OF WASHINGTON
WASHINGTON STATE PATROL
WASHINGTON STATE TOXICOLOGY LABORATORY
2203 Airport Way South, Suite 360 • Seattle, Washington 98134-2027 • (206) 262-6100 • FAX (206) 262-6145

January 6, 2023

Washington Association of Prosecuting Attorneys
206 10th Ave SE
Olympia, WA 98501

Subject: UPDATE 2022 Fourth Quarter NIST Environmental Results

Follow-up sample collection was completed on December 21, 2022. Results from the resampling were received on January 4, 2023 and no compounds were detected. The area was placed back in-service January 6, 2023.

A copy of the NIST results are attached and will be posted to the Toxicology Laboratory website: https://wsp.wa.gov/forensics/toxicology.htm

Sincerely,

Elizabeth Gough
Toxicology Laboratory Division Commander

EG:eg
Attachment

January 6, 2023

Participation in NIST Study (Quarterly Environmental Sampling) – Narcotics Background Quantitation & Screening Summary Report

The Toxicology Laboratory continues its collaboration with NIST in a current study, the goal of which is to establish drug background levels present in a forensic science facility. NIST provides the Laboratory with test kits, which the Laboratory uses to collect environmental samples, and the samples are sent to NIST for testing.

Follow-up sample collection from the area with a detectable result from the November 2022 round of environmental sampling was performed 12/21/22. The sample was sent to NIST for analysis and inclusion in the study. A summary of testing performed by NIST is attached (report dated 1/4/23), with test results listed on page 4 of the report. The sample (identified as Bay 9 Under Window) had no compounds detected when screened by DART-MS and no quantifiable levels of any compound in the LC-MSMS panel.

The regularly scheduled full round of environmental sampling, to include additional samples collected from the Bay 9 area, will take place in first quarter 2023.

January 4th, 2023

Eric Lo
Laboratory Manager
Washington State Patrol
2203 Airport Way South
Seattle, WA 98134

Brian,

Thank you for participating in our study. The goal of this project was to establish the narcotics background present in a forensic science laboratory. The following report contains the results from the analysis of 1 sample collected from the Washington State Toxicology Laboratory on December 21, 2022. The analysis scheme involved a broad screening of over 1100 drugs and common excipients and a targeted quantification of 29 drugs.

We would be happy to discuss these results in further detail with you at any time and hope to continue collaborative efforts in the future. If we can be of any assistance to you, please don't hesitate to ask.

Sincerely,

Edward Sisco

Research Chemist
Materials Measurement Science Division
National Institute of Standards & Technology
100 Bureau Dr. Gaithersburg, MD 20899
Phone: (301)975-2093
E-mail: edward.sisco@nist.gov

Narcotics Background Quantitation & Screening Summary

Introduction

The recent spike in forensic cases containing highly toxic fentanyl analogues highlights the critical need to safeguard analysts from inadvertently encountering these, or other, compounds through skin adsorption and/or inhalation.[1] Establishing background levels of compounds of interest in a forensic laboratory can provide drug analysts and laboratory quality managers with valuable information to make informed decisions on a range of topics such as: workflow processes, adequate PPE, cleaning protocols, and occupational safety hazards.

Given that trace amounts of narcotics have been reported in a variety of environments including public spaces,[2] and that instruments continue to improve in sensitivity, it is important to monitor the environmental background levels of these compounds. For field and/or screening applications, establishing the background is key to setting instrument detection thresholds and preventing false positives.[3] This is especially critical in environments where there is an expected higher background level such as prisons or border crossings. In a laboratory setting, high environmental background levels can suggest a need to monitor background for quality and health purposes.

Finally, since forensic laboratories continue to struggle with a high number of emerging drug cases and rising backlogs, opportunities for rapid screening / presumptive testing are desired. The ability to screen evidence in a high throughput manner with little to no sample preparation is currently being investigated. To ensure the results from such analysis are from the evidence and not from possible background within the laboratory, a baseline of the environment must be known.

Experimental

Samples were collected with manual Nomex wipes (Part No. DSW1210P) (DSA Detection, North Andover, MA) which are commonly used for particle collection in trace contraband detection. The particle collection efficiency of this material has been previously measured by our laboratory and results demonstrate that it is an adequate substrate for the collection of trace residues off a variety of surfaces.[4] A total of 1 sample was provided to us for analysis. Upon receipt samples were stored, at -10 °C, until they were processed.

Prior to analysis, the Nomex wipes were trimmed in size to remove the unused area of the wipe. The trimmed wipe was placed in a 10 mL amber glass vial and extracted with 4.0 mL of methanol (Chromasolv Grade, Sigma-Aldrich). The 4.0 mL extract was subsequently split into two 2.0 mL aliquots – one for the screening analysis and one for the quantitation analysis. Both aliquots were then evaporated to dryness under a stream of nitrogen. The aliquot for the screening analysis was reconstituted in 200 µL of acetonitrile, to concentrate the sample, while the aliquot for quantitation was reconstituted in 500 µL of methanol containing 5 internal standards. The quantitation aliquot was directly loaded onto the LC-MS/MS system.

Chemicals & Materials

Analytes for the screening and quantitation studies were obtained from either Cayman Chemical (Ann Arbor, MI), Cerilliant (Round Rock, TX), or Sigma-Aldrich (St. Louis, MO) as 1 mg/mL standards (when possible) or as pure crystalline material. Solvents for extraction and the LC

mobile phase were Chromasolv-grade solvents purchased from Sigma-Aldrich. For quantitation, the 5 deuterated internal standards were: methamphetamine-d5, heroin-d9, cocaine-d3, fentanyl-d5, and THC-d9. They were added to 1 L of methanol, providing an internal standard concentration of approximately 1 µg/mL, to be used for the reconstitution of the quantitation aliquot. Wipe materials were purchased from Smiths Detection and used as-is.

Quantitation of Drugs by LC-MS/MS

In order to have the highest level of sensitivity and specificity for the quantitation runs, a LC triple quadrupole MS operating in multiple reaction monitoring (MRM) mode was used. The system consisted of a Thermo Ulti-Mate 3000 LC system coupled to a ABSciex Q-Trap 4000 mass spectrometer. Separation was achieved using a Restek Raptor Biphenyl column (150 mm x 4.6 mm x 2.7 µm). The analysis time was 15 minutes with a flow rate of 0.75 mL/min and an injection volume of 15 µL. During the run, a 12-minute solvent gradient was used (95 % water / 5 % methanol + 0.1 % formic acid to 100 % methanol with 0.1 % formic) followed by a 3-minute isocratic period (100 % methanol + 0.1 % formic acid). The MS utilized zero-air nitrogen as both the desolvating and nebulizing gases. An electrospray ionization (ESI) source was used with a temperature of 550 °C and a spray voltage of +5500 V. A timed MRM was used to monitor two transitions for all drugs (one for quantitation and one for confirmatory identification) and one transition for each of the 5 internal standards. The MRM detection window was set to 120 s and the target scan time was set to 0.1 s.

Quantitation was calculated by taking the ratio of the peak areas of a drug to the appropriate internal standard and comparing that ratio to a 13-point calibration curve. Absolute concentrations reported in the summary account for the various dilution and sample splitting steps in the extraction process. They do not, however, account for the extraction efficiency of the Nomex wipe, which is typically in the range of 30 % - 40 %.

Screening of Drugs by DART-MS

The aliquot prepared for the screening analysis was sampled using glass microcapillary rods. The DART-MS system used a JEOL AccuTOF JMS T100-LP time-of-flight mass spectrometer (JEOL USA) coupled with a DART ion source (IonSense). A 400 °C DART gas temperature, a +50 V DART exit grid voltage, and helium as the ionization gas was used. Mass spectrometer settings included operation in positive ionization mode, a +800 V peaks voltage, a +5 V orifice 2 and ring lens voltage, and a mass scan range of 80 m/z – 800 m/z. To obtain characteristic molecular and fragmentation spectra, the orifice 1 voltage was cycled between +30 V and +60 V.

PEG-600 was used as a mass calibrant and AB-FUBINACA was used as a mass drift compensation compound. The resulting mass spectra were searched against an in-house created library of over 1,100 compounds using the NIST DART-MS Data Interpretation Tool. The screening results reported met the following identification criteria: the protonated molecular ion peak of the compound was present at greater than 5 % relative abundance and within ±5 amu of the calculated accurate mass.

Results

There were no quantifiable levels of any compound in the LC-MS/MS panel found in the sample provided (labelled “Bay 9 Under Window”).

From the presumptive screening analysis, no compounds of interest were detected in the sample provided, when analyzed by DART-MS utilizing a 5 % relative intensity identification threshold.

As stated in the opening letter, we would be more than happy to discuss these results with you and other interested members of your lab. If you would like us to analyze samples from additional areas, re-sample after any operational changes, or re-sample to monitor trends, we would be happy to do so. If there is any other way which we could be of assistance or form a stronger collaboration, please let us know.

Disclaimer

Certain commercial equipment, instruments, or materials are identified in this document. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

References

1. Daughton, C. G. Illicit Drugs and the Environment. in *Illicit Drugs in the Environment* (eds. Castiglioni, S., Zuccato, E. & Fanelli, R.) 1–27 (John Wiley & Sons, Inc., 2011).

2. Forbes, T. P. & Najarro, M. Ion mobility spectrometry nuisance alarm threshold analysis for illicit narcotics based on environmental background and a ROC-curve approach. *Analyst* **141,** 4438–4446 (2016).

3. Sisco, E. *et al.* Rapid detection of fentanyl, fentanyl analogues, and opioids for on-site or laboratory based drug seizure screening using thermal desorption DART-MS and ion mobility spectrometry. *Forensic Chem.* **4,** 108–115 (2017).

4. Verkouteren, J. R. *et al.* A method to determine collection efficiency of particles by swipe sampling. *Meas. Sci. Technol.* **19,** 115101 (2008).

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November 23, 2022

Washington Association of Prosecuting Attorneys
206 10th Ave SE
Olympia, WA 98501

Subject: 2022 Fourth Quarter NIST Environmental Results

The Washington State Patrol Toxicology Laboratory continues its participation in a NIST Study, the goal of which is to establish drug background levels present in a forensic science facility. Results from fourth quarter 2022 environmental sampling[1] were received on November 17, 2022. One sample had no compounds detected when screened by DART-MS, but had a detectable level of oxycodone when analyzed by LC-MSMS.

The area with a detectable level of oxycodone is not used for casework sampling or processing; however, the area was cleaned by laboratory personnel and placed out of service on November 17, 2022. Initial evaluation showed adjacent areas, where sample preparation is performed, were included in 2022 third quarter sampling, with no detected compounds. Laboratory casework testing was reviewed and no opiates confirmation testing has been performed in the adjacent area since December 2019. A targeted review of screening casework performed in the adjacent area was also completed, with reported results meeting laboratory criteria. Following the preliminary review of casework, there is no indication of impact to casework at this time.

A copy of the NIST results are attached and will be posted to the Toxicology Laboratory website: https://wsp.wa.gov/forensics/toxicology.htm

Sincerely,

Elizabeth Gough
Toxicology Laboratory Division Commander

EG:ab/eg/el
Attachment

[1] Samples were collected on November 8, 2022

November 23, 2022

Participation in NIST Study (Quarterly Environmental Sampling) – Narcotics Background Quantitation & Screening Summary Report

The Toxicology Laboratory continues its collaboration with NIST in a current study, the goal of which is to establish drug background levels present in a forensic science facility. NIST provides the Laboratory with test kits, which the Laboratory uses to collect environmental samples, and the samples are sent to NIST for testing.

In accordance with the Laboratory's quarterly environmental sampling plan, Toxicology Laboratory personnel collected samples on 11/8/22, which the Laboratory sent to NIST for analysis. A summary of testing performed by NIST is attached (report dated 11/17/22), with test results listed on page 4 of the report. Of the 25 samples submitted for analysis, one sample (identified as Sample #8) was negative when screened by DART-MS but had a detectable level of oxycodone when analyzed by LC-MSMS.

The area with a detectable level of analyte, and adjacent areas, were cleaned by laboratory personnel on November 17th. Follow-up sample collection from those areas using NIST test kits is planned, with samples to be sent to NIST for analysis and inclusion in the study.

The next regularly scheduled full round of environmental sampling is planned for first quarter 2023.

November 17th, 2022

Brian Capron
Acting Laboratory Manager
Washington State Patrol
2203 Airport Way South
Seattle, WA 98134

Brian,

Thank you for participating in our study. The goal of this project was to establish the narcotics background present in a forensic science laboratory. The following report contains the results from the analysis of 25 samples collected from the Washington State Toxicology Laboratory on November 8, 2022. The analysis scheme involved a broad screening of over 1100 drugs and common excipients and a targeted quantification of 29 drugs.

We would be happy to discuss these results in further detail with you at any time and hope to continue collaborative efforts in the future. If we can be of any assistance to you, please don't hesitate to ask.

Sincerely,

Edward Sisco

Research Chemist
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Results

From the 25 samples provided, listed in Table 1, one was found to have quantifiable levels of a surface. Sample #8 (Bay 9 Bench) had a detectable level of oxycodone (0.11 µg). No other sample had detectable levels of any of the 29 drugs included in the LC-MS/MS panel.

From the presumptive screening analysis, no compounds of interest were detected in any of the samples provided, when analyzed by DART-MS utilizing a 5 % relative intensity identification threshold.

As stated in the opening letter, we would be more than happy to discuss these results with you and other interested members of your lab. If you would like us to analyze samples from additional areas, re-sample after any operational changes, or re-sample to monitor trends, we would be happy to do so. If there is any other way which we could be of assistance or form a stronger collaboration, please let us know.

Table 1. Locations of samples collected. All samples were noted to have been collected on November 8, 2022.

Sample #	Location	Sample #	Location
1	Hood #4 Benchtop	14	Volatile Fridge Door
2	Hood #4 Sash	15	LC/MS #2 Bench
3	Hood #10 Benchtop	16	GC/MS #5 Bench
4	Hood #10 Sash	17	QQQ #4 Bench
5	Bay ½ Bench	18	GC/MS #14 Bench
6	Bay 5/6 Bench	19	HSGC #7 Bench
7	Bay 7/8 Bench	20	Refrigerator #15 (door)
8	Bay 9 Bench	21	DI Water System
9	Standards Freezer Door	22	Extinguisher B (Door)
10	Flammable Cabinet (Hexanes)	23	Vacant Desk (AM)
11	BA#1 Benchtop	24	Bench (Cubiles up front)
12	BA#2 Benchtop	25	JS Cabinet
13	Peak H (alcohol)		

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References

1. Daughton, C. G. Illicit Drugs and the Environment. in *Illicit Drugs in the Environment* (eds. Castiglioni, S., Zuccato, E. & Fanelli, R.) 1–27 (John Wiley & Sons, Inc., 2011).

2. Forbes, T. P. & Najarro, M. Ion mobility spectrometry nuisance alarm threshold analysis for illicit narcotics based on environmental background and a ROC-curve approach. *Analyst* **141,** 4438–4446 (2016).

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