

September 19, 2023

UPDATED - NIST Study (Quarterly Environmental Sampling) 2023 Quarter Two (Q2)

On August 17, 2023, the Toxicology Laboratory received an updated letter and an updated summary report from NIST for the 2023 Q2 sampling, following a request for correction. The updated letter and updated summary report dated August 17, 2023 included corrections to the recipient name (page 1) and corrected the number of samples (page 2) to accurately reflect the number of samples received by NIST for the Q2 sampling.

June 15, 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.

In accordance with the Laboratory's quarterly environmental sampling plan, Toxicology Laboratory personnel collected samples on 5/18/23, which the Laboratory sent to NIST for analysis. A summary of testing performed by NIST is attached, with test results listed on page 4 of the report.

The next round of environmental sampling is planned for third quarter 2023.

August 17th, 2023

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

Eric,

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 results for the 25 samples collected by the Washington State Toxicology Laboratory on May 18th, 2023. 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
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Narcotics Background Quantitation & Screening Summary

Introduction

The recent spike in forensic cases containing highly toxic fentanyl analogs highlights the critical need to safeguard analysts from inadvertently encountering these, or other, compounds.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 including workflow processes, adequate PPE, cleaning protocols, and occupational safety hazards.

Given that trace amounts of illicit drugs 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 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 data quality and personnel 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 25 samples were provided to us for analysis. Upon receipt, samples were stored at -10 °C until they were processed.

Prior to analysis, 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. The aliquot for the screening analysis was reconstituted in 200 μ L of acetonitrile, to concentrate the sample, while the aliquot for quantitation was reconstituted in 500 μ L 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 an 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 min with a flow rate of 0.75 mL/min and an injection volume of 15 µL. During the run, a 12-min solvent gradient was used (95 % water / 5 % methanol + 0.1 % formic acid to 100 % methanol with 0.1 % formic) followed by a 3-min isocratic period (100 % methanol + 0.1 % formic acid). The MS utilized zero-air nitrogen as both the desolvating and nebulizing gas. An electrospray ionization (ESI) source was used with a temperature of 550 °C and a spray voltage of +5500 V. A timed MRM method 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 were 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 m/z 80 to m/z 800. 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 mmu of the calculated accurate mass.

Results

None of the 25 provided samples (Table 1) were found to contain a quantifiable level of any compound in the LC-MS/MS panel.

None of the 25 provided samples (Table 1) were found to contain a detected level of any compound in the DART-MS screening method.

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 May 18th, 2023.

Sample #	Location	Sample #	Location
1	SE I.R. Counter 2	14	Balance Room Door Handle
2	SE I.R. Counter 4	15	Fridge 14 Door Handle
3	SE I.R. Counter 5	16	Freezer 9 Door Handle
4	NE I.R. Counter 2	17	Autosampler Vial Storage Cabinet
5	NE I.R. Counter 3	18	DI H2O System Nozzle
6	NE I.R. Counter 5	19	Counter Outside Vault
7	Counter by BA Hood 2	20	"Toxicology Laboratory" Cart
8	Inside BA Hood 2	21	Pillar in Bay 1
9	SE Lab Exit Door Interior Handle	22	Eyewash #1 Paddle
10	Alcohol Prep Counter	23	Counter Centrifuge Bay 1
11	South Floor Centrifuge	24	Glove Dispenser Hood 6
12	Sink Handle Bay 11	25	Fire Extinguisher B Handle
13	MeOH Flammable Cabinet Handle		

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).

June 13th, 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 25 samples collected from the Washington State Toxicology Laboratory on May 18th, 2023. The analysis scheme involved a broad screening of over 1100 drugs and common excipients and a targeted quantification of 29 drugs.

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Sincerely,

Edward Sisco

Research Chemist
Materials Measurement Science Division
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100 Bureau Dr. Gaithersburg, MD 20899
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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.

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References

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