March 6, 2024

Quarterly Environmental Sampling – Narcotics Background Quantitation & Screening Summary Report

The Toxicology Laboratory continues its collaboration with NIST. 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 Seattle Laboratory's quarterly environmental sampling plan, a representative of the Washington State Patrol's Safety and Wellness Team collected samples on 12/8/23, which the Laboratory sent to NIST for analysis. A summary of testing performed by NIST is attached, with test results listed on page 3 of the report.

The next round of environmental sampling is planned for the first quarter of 2024.

February 5th, 2024

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

Eric,

Thank you for participating in our study. The following report contains results for the 25 samples collected by the Washington State Toxicology Laboratory in December 2023. The goal of this project was to establish the narcotics background present in a forensic science laboratory. The analysis scheme involved a broad screening of over 1,100 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

Drug 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 Nomex wipes, purchased from Smiths Detection, and used as-is. The particle collection efficiency of this material has been previously measured by our laboratory and has been demonstrated to be adequate for the collection of trace residues off a variety of surfaces.[4] A total of 25 samples were provided for analysis. Upon receipt, samples were stored at -10 °C until they were processed.

Prior to analysis, wipes were trimmed in size to remove the unused area. The trimmed wipe was placed in a 10 mL amber glass vial and extracted with 4.0 mL of methanol (Omnisolv 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 evaporated to dryness. The aliquot for the screening analysis was reconstituted in 200 µL of acetonitrile, while the aliquot for quantitation was reconstituted in 500 µL of methanol containing an internal standard.

Chemicals & Materials

Analytical standards were obtained from either Cayman Chemical, Cerilliant, or Sigma-Aldrich as 1 mg/mL solutions (when possible) or as crystalline material. For quantitation, a single internal standard of tetracaine HCl was used. One milligram of tetracaine HCl was added to 1 L of methanol, providing an internal standard concentration of approximately 1 µg/mL.

Quantitation of Drugs by LC-MS

An liquid chromatography (LC) triple quadrupole mass spectrometer (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 MS. 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 a 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 (one for quantitation and one for confirmatory identification) for all drugs and the internal standard. 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 internal standard and comparing that ratio to a multi-point calibration curve. Absolute concentrations, if reported in the summary, account for the various dilution and sample splitting steps in the extraction process. The concentrations do not account for the extraction efficiency of the wipe, which is surface dependent but is typically in the range of 30 % to 40 %.

Screening of Drugs by DART-MS

Screening was completed by dipping a glass microcapillary rod into a solution and analyzing it by direct analysis in real time mass spectrometry (DART-MS). A JEOL AccuTOF JMS T100-LP time-of-flight MS (JEOL USA) coupled with a DART ion source (Bruker Daltonics) was used. A 400 °C DART gas temperature, +50 V DART exit grid voltage, and helium source gas were used. The mass spectrometer was operated in positive ionization mode with a +800 V peaks voltage, +5 V orifice 2 and ring lens voltage, and a mass scan range of *m/z* 80 to *m/z* 800. To obtain molecular ion 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. Compound identification required the following identification criteria: the protonated molecular ion or base peak of the compound must be 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 panel.

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

Table 1. Locations of samples collected.

Sample #	Location	Sample #	Location
1	Inst. Rm Table 1	14	Bay 5 Counter
2	Inst. Rm Table 2	15	Hood 7 Counter
3	Inst. Rm Table 4	16	Hood 8 Counter
4	Inst. Rm Table 5	17	Hood 5 Counter
5	Inst. Rm Table 3	18	Hood 6 Counter
6	Hood BA2	19	Hood 3 Counter
7	Bench 1 Next to BA2	20	Hood 4 Counter
8	Bench 2 Next to BA2	21	Hood 1 Counter
9	Bench Next to BA1	22	Worklist Counter
10	Hood BA1	23	Olympus Counter
11	Hood 11 Counter	24	Evidence Ckout Counter
12	Across from Hood 11	25	Island Outside Vault
13	Hood 9 Counter		

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).