Trace explosives sampling for security applications (TESSA) study: Evaluation of procedures and methodology for contact sampling efficiency

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ABSTRACT

The detection of trace amounts of explosive materials is critical to the security at mass transit centers (*e.g.*, airports and railway stations). In a typical screening process, a trap is used to probe a surface of interest to collect and transfer particulate residue to a detector for analysis. The collection of residues from the surface being probed is widely viewed as the limiting step in this process. A multi-institutional study was performed to establish a methodology for the evaluation of sampling media collection efficiencies. Dry deposited residues of 1,3,5-trinitroperhydro-1,3,5-triazine (RDX), C-4 (an RDX-based explosive), and pentaerythritol tetranitrate (PETN) were harvested from acrylonitrile butadiene styrene (ABS) plastic, ballistic nylon (NYL), and uncoated aluminum surfaces using muslin, Texwipe cotton, and stainless-steel mesh traps. Transfer and collection efficiencies of the sample media were calculated based on liquid chromatography-mass spectrometry analysis. Dry transfer efficiencies (DTE%) to all tested surfaces were greater than 75%, with transfer to ABS plastic being the lowest. Collection efficiency (CE%) varied significantly across the traps and the surfaces, yet some conclusions can be drawn; nylon had the lowest CE% for all cases (~10%), and the stainless steel mesh had the lowest CE% for the evaluated traps (~20%). Though the testing parameters have been standardized among the participants to establish a framework for an independent comparison of contact sampling media and surfaces, substantial variations in the DTE% and the CE% were observed, suggesting that other variables can affect contact sampling.

1. Introduction

The ability to harvest residual explosives from surfaces is crucial for detecting these threats in security applications. Trace amounts of microscopic particles adhere to the hands and equipment of those handling explosives, explosive precursors, or other illicit substances. These residues can be subsequently transferred by contact to clothing, baggage, parcels, vehicles, and other surfaces. Among the challenges in trace detection is developing effective methods to harvest a residue with a wide variety of physicochemical properties from different types of surfaces over a range of environmental conditions. Prior work has shown that a detectable level of explosive residue is transferred to surfaces from hands following the handling of bulk explosives [1,2]. Verkouteren et al. showed that RDX particles from C-4 residue could be detected on a glass surface even after 50 consecutive fingerprints [1]. Transfer directly onto substrates for analytical testing has also been presented by Tam et al. (2013) when C-4, Detasheet (a PETN-based explosive), Semtex-H (an RDX and PETN-based explosive), 2,4,6-trinitrotoluene (TNT), and hexamethylene triperoxide diamine (HMTD) residues were directly transferred onto several surfaces before quantification [3].

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Table 1
Summary of studies [9,14–16] of the performance of contact sampling of trace explosive residues.

	Analyte	Deposition	Sample Preparation	Substrate	DTE (%)	SD (%)	Trap Material	CE (%)	SD (%)
Song-im et al. (2012)	RDX	15 µg	Aliquot of sample (Concentration of 1 µg/µL) deposited directly onto the surface and allowed to dry for a minimum time of 4 min.	Glass	N/A	N/A	Cotton	75.2	6.4
							Polyester	78.9	2.4
				Plastic			Cotton	73.0	15.7
							Polyester	86.0	3.6
	TNT			Glass			Cotton	77.4	10.9
							Polyester	70.6	8.6
				Plastic			Cotton	50.1	17.5
							Polyester	20.1	19.5
	PETN			Glass			Cotton	91.6	1.1
							Polyester	86.1	4.2
				Plastic			Cotton	58.7	5.4
							Polyester	46.7	22.6
	TATP			Glass			Cotton	32.0	18.4
							Polyester	48.4	16.5
				Plastic			Cotton	33.8	11.6
							Polyester	10.8	6.0
Staymates et al. (2016)	RDX	N/A	Inkjet-printed RDX samples. Mass not reported for security purposes.	Canvas	N/A	N/A	Nomex	16.8	7.8
							TCFG	6.2	4.5
				Clean Cardboard			Nomex	15.7	18.0
							TCFG	1.7	0
				Dusty Cardboard			Nomex	6.6	2.8
							TCFG	7.1	3.9
Fisher et al. (2017)	RDX	1 µg	Direct Deposition (Mass was not reported)	Glass	N/A	N/A	Muslin	82.5	12.5
							Nomex	72.0	15.0
							TCFG	2.0	0.5
	HMX						Muslin	80.2	5.3
							Nomex	78.2	4.6
							TCFG	0.0	0.0
Robinson et al. (2018)	RDX	350 ng	8 by 8 array on foil-based PTFE using DOD inkjet printing. Dry deposition with deposition length 2–3 cm	ABS Plastic	66.7	12.6	Nomex	43.8	5.6
				Ballistic Nylon	93.9	5.3		10.3	1.8
				Cardboard	93.9	4.3		20.7	2.0
				Packaging Tape	24.5	9.6		31.8	12.6
				Roughened Tape	91.7	4.8		31.2	6.3
				Synthetic Leather	97.9	3.2		29.3	5.4
				Steel	91.5	4.1		34.5	2.6
DeGreeff et al. (2019)	RDX	10 µg	Direct Deposition (Mass was not reported)	Kraft Paper	95.2	3.6	Nomex	24.8	14.3
							TCFG	1.9	0.5
				Cordura	94.4	4.5	Nomex	35.3	8.8
							TCFG	1	0.9
	TNT	1 µg		Kraft Paper	60.6	4.2	Nomex	11	5.7
							TCFG	3.2	2.2
				Cordura	68.4	14.3	Nomex	11.2	4.2
							TCFG	2.5	1.7
	C-4	N/A		Kraft Paper	N/A	N/A	Nomex	0.63	0.6
							TCFG	0.61	0.6
				Cordura	N/A	N/A	Nomex	25	5.2
							TCFG	3.2	0.6

*Teflon-coated fiberglass (TCFG); Polytetrafluoroethylene (PTFE); Standard Deviation (SD).

The current harvesting methods rely on the swabbing of target surfaces using a sampling trap. This method employs downward pressure and frictional forces to remove particles of interest from the sampled surface. The capture of a particle can be considered as a simple balance of forces. For a particle to be removed from a surface, adhesion forces between the particle and surface must be overcome by forces between the particle and the sampling trap. The forces involved in the sampling include van der Waals, electrostatic, capillary, and frictional forces [4–6]. The adhesion forces are affected by the surface, trap and analyte composition and morphology [6]. In current practice, the traps (the term "swabs" or "swipes" are used interchangeably) are analyzed in explosive trace detectors (ETDs). When the trap with collected residue is introduced into the ETD system, the residue is thermally desorbed, and the molecular vapors are delivered to an ion mobility spectrometer (IMS) or a mass spectrometer (MS) for separation and detection.

Previous studies have investigated factors affecting harvesting efficiency, including swiping force and speed, types of analyte, trap and surface materials, and particle size [3,6–11]. While the prior studies provide some insight into contact sampling efficacy, they differ in the methods used for sample preparation, loading levels, test conditions, and sampling methodology. In particular, sample preparation significantly affects particle distribution over a surface, biasing the interfacial configuration, and plays an essential role in particle adhesion and capture. There is a need for a quantitative approach for evaluating harvesting efficiencies of commercial traps using a standardized methodology. Table 1 summarizes the quantitative harvesting efficiencies reported in previous studies for common explosives and surfaces. Song-im et al. [12] used cotton and polyester traps with solvent for organic and inorganic explosives residues. The highest recovery for a range of target compounds, including RDX, TNT, PETN, triacetone triperoxide (TATP), chlorates, and nitrates, was achieved by swabbing with traps wetted with acetonitrile. While solvent-based protocols were found effective, the ETD-based screening utilizes dry swabs to avoid chemical interference with the detector. Fisher et al. [9] and Staymates

Table 2
Summary of round-robin contact sampling testing.

Residue	Surface	Trap	Partner	Replicates/Partner[a]	Number of Experiments
RDX	AL, ABS, NYL	SS, TX, MLN	PUR, NMT, UW	10	270
C-4	AL, ABS, NYL	SS, TX, MLN	PUR, NMT, UW	10	270
PETN	AL, ABS, NYL	SS, TX, MLN	PUR, NMT, UW	10	270
				Total (N)	810

[a] Replicates/partner = number of replicates for each residue, surface, and trap at each partner institution.

et al. [10] investigated the effect of trap re-use on CE%. Both studies used scanning electron microscopy (SEM) to evaluate changes in trap morphology with reuse. An increase in CE% was noted for rough or fibrous traps with long unsupported fibers. The results can be explained by an increase in total surface area for particle binding on rougher traps or a greater chance of entanglement for fibrous traps. While both studies agreed that CE% was improved with repeated use, Fisher et al. showed a decline in CE% after 5–10 successive uses; in comparison, Staymates et al. indicated a continued increase in particle CE% after as many as 1000 uses. The discrepancy between the two studies could be due to the type of particles harvested and the swabbing conditions used in the investigations. Staymates et al. used Polystyrene Latex (PSL) spheres as surrogates for explosive residues under controlled laboratory conditions, while Fisher et al. used RDX and HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) particles in more realistic but relatively uncontrolled conditions of an airport screening scenario.

The sampling standardization protocols have been under development by the National Institute of Standards (NIST). Verkouteren et al. [13] reported a method for measuring the CE% of trace explosive particles based on an existing test method, i.e., "Static and Kinetic Co-efficients of Friction of Plastic Film and Sheeting" (ASTM D1894-08). The standardized approach allows focusing on comprehensive investigations of individual parameters in the wipe-sampling process: the trap and test surfaces, the swabbing distance, the force of collection (applied load), and the swabbing pattern. More recently, Robinson et al. addressed the effect of applied load, swabbing distance, and sampling pattern on harvesting efficiency of RDX from various surfaces [14]. In contrast to the trends of improved CE% for rougher traps, the authors reported that CE% from rougher and fibrous test surfaces (e.g., fabrics, cardboard, and wood) is lower than from the smooth surfaces, which can be attributed to residues becoming embedded in the fibrous matrix or by residue re-deposition in features on the surface during swabbing. DeGreeff et al. studied the effects of repeated trap use on the trap's surface texture changes and its effectiveness in collecting explosives residues [15]. Surface textures (roughness and hairiness) were evaluated using silhouette microscopy based on the techniques and metrics used in the textiles industry. The texture measurements correlated well with the CE%; the increased wear leads to higher collection efficiencies.

However, the CE% can be influenced by other factors, making it difficult to establish specific causalities.

While the prior studies provide some insight into contact sampling efficacy, they differ in the methods used for sample preparation, loading levels, test conditions, and sampling methodology. In particular, sample preparation significantly affects particle distribution over a surface, biasing the interfacial configuration, and plays an essential role in particle adhesion and capture. There is a need for a quantitative approach for evaluating harvesting efficiencies of commercial traps using a standardized methodology.

This work aims to build upon a testing procedure previously reported by NIST [13] to develop a methodology that eliminates user bias, allowing for direct comparison between contact sampling materials. The new methodology was evaluated in a multi-institutional study using three different explosives, three types of traps, and three different surfaces. During the round-robin study, three universities performed contact sampling tests using a nominally identical protocol, and the collected samples were analyzed by a third-party analytical laboratory.

2. Materials and methods

Removal of RDX, C-4, and PETN residues from AL, ABS, and NYL surfaces using MLN, Texwipe cotton, and SS traps was studied. The sampling work was performed in a round-robin fashion at Purdue University (PUR), the University of Washington (UW), and New Mexico Tech (NMT), following the methods outlined above. A quantitative assessment of the sampling effectiveness was performed by Signature Science, LLC (Austin, TX). Table 2 summarizes the experiments conducted by each university.

2.1. Wipes and test surfaces

Three model trap materials were used as received, including textured cotton wipes (1) Texwipe TX304 Alphawipe, ITW; Texwipe, NC, USA), muslin wipes (MLN: SSW5883P, DSA Detection, MD, USA), and stainless-steel mesh (SS: Dutch twill wire cloth, 316 LSS ANSI composition; L3 Security and Detection Systems, FL, USA). Test surfaces included uncoated Aluminum (AL: 3003H14 06X06X025 with MILL OIL, Model Number 60696, ACT Test Panels, MI, USA), ballistic nylon fabric (NYL: 1000 Denier Nylon Cordura, SKU:100235-BK-59/60IN-DUC, Top Value Fabrics Carmel, IN), and ABS plastic with textured hair-cell finish (ABS: SKU: ABSBLK0.125HC48X48, Ridout Plastics, San Diego, CA).

2.2. Sampling instrument

A modified standard operating procedure reported by Verkouteren et al. [13] was used to establish repeatability in the contact sampling. As shown schematically in Fig. 1, a slip peel tester (Imass TL-2200, Accord, MA, USA) with the configuration based on ASTM D1894-08 was used. A modification included the addition of bracket mounts to secure test substrates to the stage. Double-sided tape was used to secure fibrous and deformable substrates to the stage. The sled mass selection was
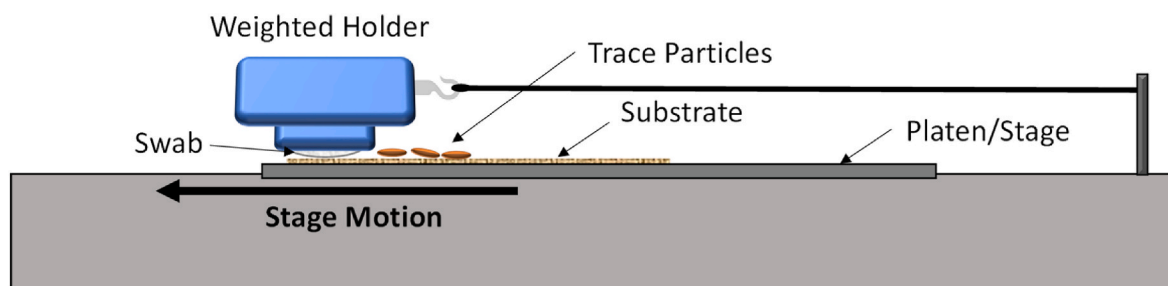

Fig. 1. Schematic of slip peel tester for contact sampling. The stage moves from right to left at 100 mm/s, and the weighted holder (sled) stays in place with the applied mass on the top — the sled's mass is 660 g.
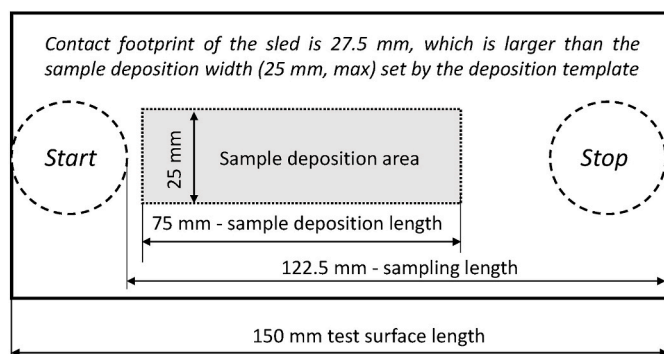
Fig. 2. Diagram of the sampling setup, top view. The contact sampling area is larger than the sample deposition area.

motivated by Robinson et al. [14], who reported that no significant improvement in CE% was observed when applied loads exceeded 660 g (~6.5 N) during sampling, except when from harvesting of NYL, where the collection improved by a factor of 1.75 at the highest tested load (1060 g). In this present work, a sled with a mass of 660 g was used. The sleds for all performers were fabricated by NIST; details on the design can be found in Ref. [13]. The stage's translational speed under the stationary sled was set to 100 mm/s, and the sampling distance was 122.5 mm.

To ensure that the entire deposited sample is exposed to the swab, the swiping area was set to be greater than the sample deposition area (described in the following section). The diagram with dimensions and sampling area is shown in Fig. 2. At the beginning of the test, the sled is resting "Start" position; during sampling, the stage is moved from right to left, with the sled ending at the "Stop" position.

2.3. Sample preparation

The Department of Homeland Security (DHS) Transportation Security Laboratory (TSL) supplied the explosive threats used in this study. RDX and PETN were obtained from a commercial (Accustandard, New Haven, CT), while the RDX-based explosive, C-4, was obtained from the laboratory's repository. All explosives were used without any further purification. The solutions were diluted to the desired concentration for this study in tetrahydrofuran (Sigma Aldrich, St. Louis, MO). The prepared working solutions were subsequently analyzed using liquid chromatography to verify the solution concentration before use.

To prepare the residues using drop cast crystallization, an aliquot of the appropriate working solution was deposited onto a Teflon® strip [(polytetrafluoroethylene, PTFE), 1 in. × 3 in. x 0.015 in., Saint-Gobain Performance Plastics Corporation, Poestenkill, NY] in a single spot using a calibrated micropipette (Eppendorf North America, Hauppauge, NY). After deposition, the solvent was allowed to evaporate under nitrogen in a low humidity overnight, leaving a dried explosive residue on the surface. The prepared drop cast PTFE samples were subsequently packaged and shipped to each university participating in the round-robin study. Note that before performing the round-robin tests, we evaluated the loss of the particles during transport between the laboratories. Negligible material losses were observed after shipping strips with trace explosive residue between the study sites.

2.4. Test procedure

2.4.1. Preparation of surfaces

The surfaces from which contact sampling would be studied were cut into a rectangular shape measuring 75 cm × 150 cm (3 × 6 inches). The NYL and ABS surfaces' topographies are randomly oriented, so there was no preferred orientation during sample preparation or testing. The AL was purchased in large sheets that were cut to fit the sampling apparatus. The grain direction for the AL was consistent across all samples, i. e., the long axis of the test surface was in the direction of the grain. After the AL coupons were cut, they were cleaned with isopropyl alcohol (>98% purity) and allowed to dry in ambient air. To speed up the drying, the surfaces could be dusted using clean compressed air. Next, the test surfaces were placed onto an analytical balance (to control the deposition force) with a custom template mounted over each. Only the region targeted for deposition was exposed, see Fig. 3.

2.4.2. Dry transfer of explosive residue

After receiving the samples, the drop cast PTFE strips were stored in desiccators to maintain a low humidity similar to that used while drying strips. For consistent sample preparation, each university followed the TSL-developed dry transfer protocol to prepare the surfaces used in this investigation [17].

Briefly, drop cast PTFE strips loaded with explosives were rubbed onto the surface of the clean substrates (Part Number: 55,890, Hillsdale, MI) several times with moderate pressure. This process transfers the explosive material from the PTFE strip to the surface of the aluminum panel with high transfer efficiency (typically >90%), creating a heterogeneous spatial distribution of particles.

The PTFE strips loaded with explosive residue were positioned against the test surfaces resting on the balance. Using an index finger, the operator applied a downward force of 10 ± 0.5 N (see Fig. 3). The
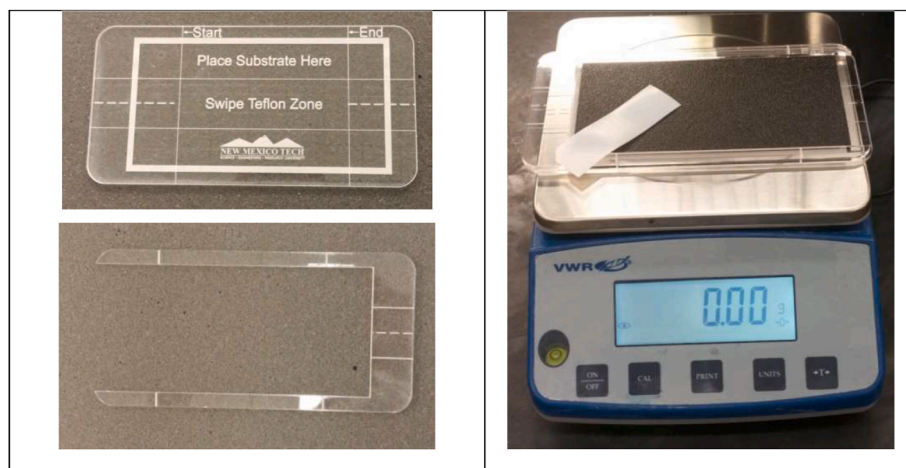

Fig. 3. Left - Template used in the dry transfer deposition; Right - Using analytical balance to control the deposition force. Though analytical balance reports mass units, the force applied to the swab is reported in grams for consistency with the previous reports, see Table 1.
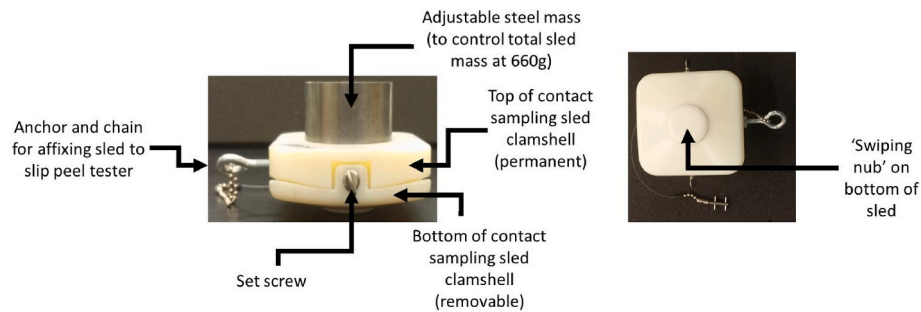
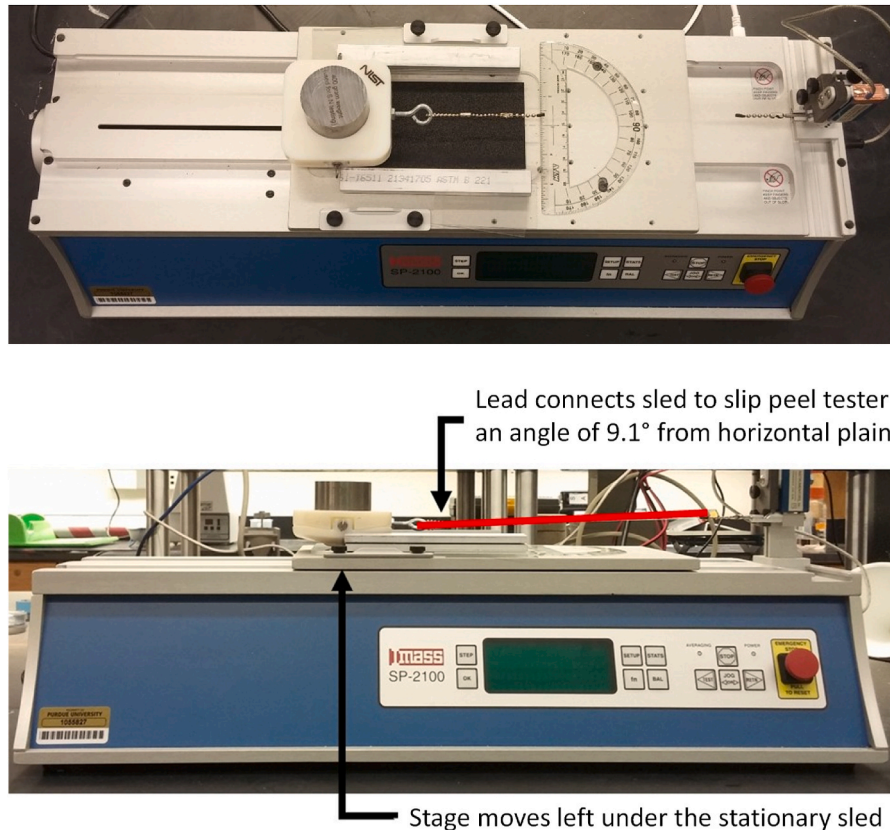**Fig. 4.** Side (left) and bottom view (right) of swiping sled.

Fig. 5. Top (top) and side (bottom) view of slip peel tester with the sled mounted on the tester's stage.

strips were dragged, under load, along the surface for 100 mm in a zigzag pattern. At all times, the zigzag motion was maintained within the template's bounds so that the residue was deposited in a confined area. This process transfers the explosive material from the PTFE strip to the surface of the aluminum panel with high transfer efficiency, creating a heterogeneous spatial distribution of particles.

2.4.3. Contact sampling of the explosive residue

The sampling sled is shown in Fig. 4. Before each experiment, the clamshell was disassembled, and aluminum foil was stretched over the 'swiping nub.' The trap of interest was subsequently placed over the foil, and the clamshell was reassembled, securing the trap and foil in the clamshell. The assembled sled was affixed to the slip peel tester's lead in the test substrate's location. The lead's angle was set at ~9° based on the lead's length and the attachment point. This angle ensured that a normal (lifting) force was not applied to the sled. A photograph of the slip peel tester with the mounted sled is shown in Fig. 5.

When the Texwipe trap was used, it was oriented so that the

herringbone pattern was in line with the sampling direction. In the experiments using muslin and SS traps, the sampling media were oriented so that the 'grain' of the trap texture was perpendicular to the swiping direction, as shown in Fig. 6. The effect of the trap pattern orientation was not studied further.

During sampling, the sled was forced to travel in a straight line over the moving stage by using 3D-printed bumpers and aluminum rails mounted on the stage (Fig. 5, top). Environmental conditions (i.e., relative humidity, temperature, and atmospheric pressure) were noted at the start of each sampling test. After swabbing, the trap and aluminum foil were removed from the holder for analysis, and the sled was cleaned thoroughly before the subsequent trial.

2.5. Residue extraction and analysis

At the end of each swiping experiment, the depleted PTFE dry transfer strip, the trap, and the aluminum foil were placed in three different 20 mL centrifuge tubes, and 2 mL of ethyl acetate were
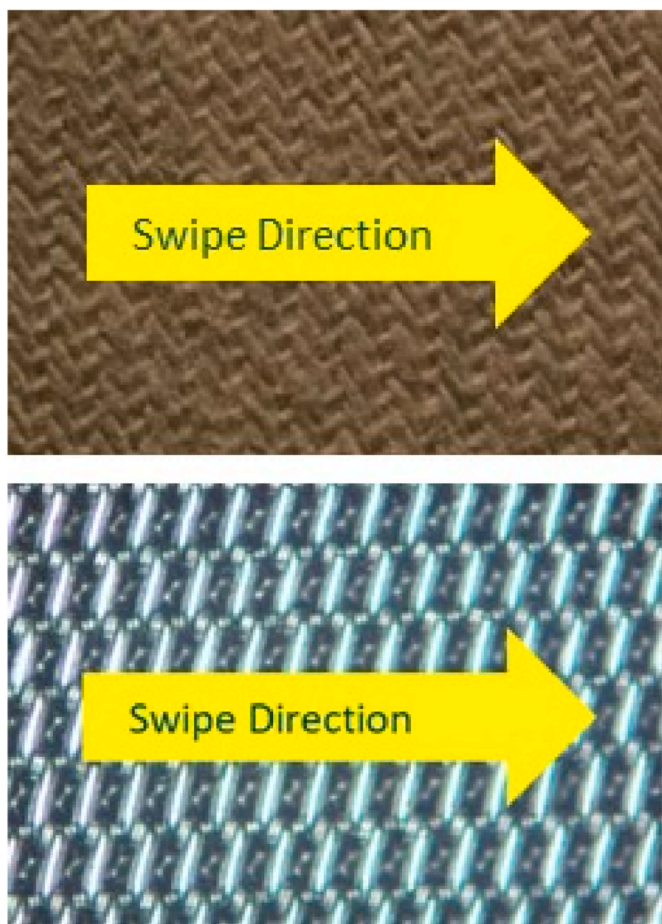
Fig. 6. Optical micrographs showing Texwipe (top) and SS (bottom) swab orientation and the swiping direction.

dispensed into each tube using a pipette. The tubes were sealed and wrapped with parafilm for storage and transport. All the samples were refrigerated at 4 °C or frozen at −18 °C if stored for longer than 24 h. The samples were subsequently shipped to a third-party analytical laboratory (Signature Science, LLC, Austin, TX, USA).

Upon receipt, each sample was dried using a nitrogen evaporator (N-EVAP) set at a flow rate of 10 L/min. The dried sample was reconstituted in 1 mL of acetonitrile containing isotopically labeled RDX as an internal standard [i-RDX ($^{13}C_3$, $^{15}N_3$) (Cambridge Isotopes, Tewksbury, MA – CNLM-7987-S)]. The reconstituted sample was then vortexed for 30 s. Approximately 100 μL of this sample was transferred to an HPLC

autosampler vial for LC-MS/MS analysis.

An Agilent model 1290 ultra-high-performance liquid chromatograph (UHPLC) coupled with an Agilent model 6410 triple quadrupole mass spectrometer (Agilent Technology, Santa Clara, CA, USA) was used to analyze all sample extracts. The analytical column was a 150 mm × 2.0 mm x 3.0 μm Gemini C18 (Phenomenex Inc. Torrance, CA, USA) with a C18 guard column cartridge and was held at 40 °C. The injection volume of samples was 10 μL. The mobile phase consisted of (A) 0.25 mM ammonium nitrate in water and (B) 0.25 mM ammonium nitrate in acetonitrile at a flow rate of 400 μL/min using a multi-step gradient (see Fig. 7).

MS/MS data were acquired in negative mode using atmospheric pressure chemical ionization (APCI) using an Agilent multimode ionization (MMI) source with the following conditions: gas temperature, 300 °C; vaporizer temperature 200 °C; gas flow, 5 L/min; nebulizer pressure, 30 psi. One multi reaction monitoring (MRM) transition was monitored for each analyte, as shown in Table 3. The collision energies and transitions were determined using the Agilent Optimizer software to determine the analyte's ionization and fragmentation performance in the final LC solvent ramp.

A multipoint calibration curve was created gravimetrically. The ratio of the instrument response versus the internal standard was used to create a regression fit for the data. This regression fit was used to quantitate the unknown samples (*i.e.,* mass found on the traps, used PTFE strips, and Al foil). In addition to the calibration standards, an internal standard blank, a substrate blank, and a calibration verification sample were also run with each sample analysis batch to ensure the stability of the internal standard and calibration curve as well as to monitor the background and verify instrument performance and cleanliness. The substrate blanks were used to ensure that background compounds did not interfere with the peaks of interest. The internal standard blanks were used to ensure the stable detection of the internal standard and identify whether or not it contributed to the overall mass quantification for all analytes.

The LC/MS analysis determined the mass of the analytes (i) remaining on the PTFE strip, (ii) collected by the trap, and (iii) by the aluminum foil for each collection experiment. Penetration through the trap was not observed, *i.e.,* aluminum foil backing did not collect any residue for any sample in this investigation. The difference between the initially spiked amount of explosive onto the transfer strip and the mass

Table 3
Mass spectrometer conditions.

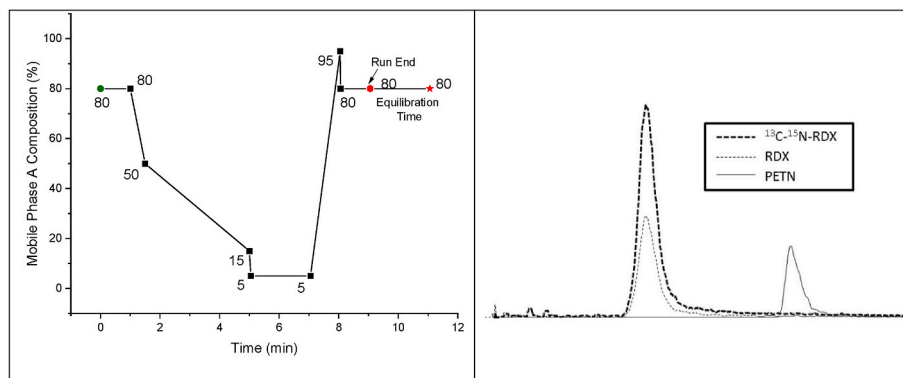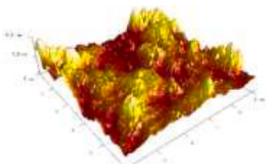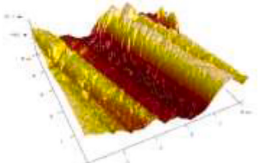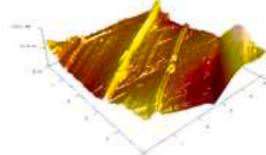| Compound Name	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Collision Energy
$^{13}C3$–$^{15}N3$-RDX	290.0	62.0	5
RDX	284.0	62.0	5
PETN	378.0	62.0	5

Fig. 7. Left – An annotated diagram of multi-step gradient procedure used to analyze the samples where the - axis indicates the mobile phase A composition only. Right – Example chromatogram from one of the calibration runs.

Table 4

Roughness of each test surface measured using AFM scans. Area scanned: 5 μm × 5 μm.

Surface	3-D AFM Surface Scans	RMS ± SD (nm)	Peak-Peak ± SD (nm)
Plastic (ABS)		27 ± 11	210 ± 76
Aluminum (AL)		44 ± 14	258 ± 131
Nylon (NYL)		87 ± 12	573 ± 60

observed on the PTFE strip after deposition was used to calculate the dry transfer efficiency (DTE) per Equation (1), where m_i is the initial mass of explosive residue deposited on the PTFE strip and m_T is the mass of explosives remaining on the PTFE strip after the dry transfer process was complete. Equation (2) illustrates how the analyte mass on the collection trap is used to determine the CE%, where m_W is the mass of explosive material collected by the traps.

$$DTE\% = \frac{(m_i - m_T)}{m_i} \times 100, \tag{1}$$

$$CE\% = \frac{m_w}{(m_i - m_T)} \times 100, \tag{2}$$

3. Results and discussion

3.1. Test surface topography

ABS, NYL, and AL were chosen as the model test surfaces for the round-robin study, as they are relevant to sampling in airport security settings and have disparate physical and chemical properties. Surface roughness measurements were made using an atomic force microscope (AFM; Bruker MultiMode 8, Veeco Nanoscope V controller, Nanoscope v1.9 software, tapping mode with RTESPTA-300 probes). Table 4 shows the root mean square (RMS) roughness (with standard deviation about the mean; SD) and peak-peak distance (the height from the tallest peak to the lowest valley) of each test surface. The topographical measurements of 15 distinct regions (5 μm × 5 μm each) were performed for each surface. The NYL is the roughest in terms of both the RMS roughness and the peak-peak distance. The larger scale (>5 μm) weave patterns contribute to the roughness parameters. The AL and ABS have similar peak-peak distances and similar RMS roughness; however, the surface patterns differ between ABS (random peaks) and AL (2D ridges).

Note that we have attempted to perform swab nanoscale texture analysis; however, the traps were so macroscopically rough that measurement was effectively impossible. The traps were highly deformable and had large voids, fibrous surface features with 10s–100 s μm height variations. From this perspective, it was not possible to differentiate the roughness of any trap from the others.
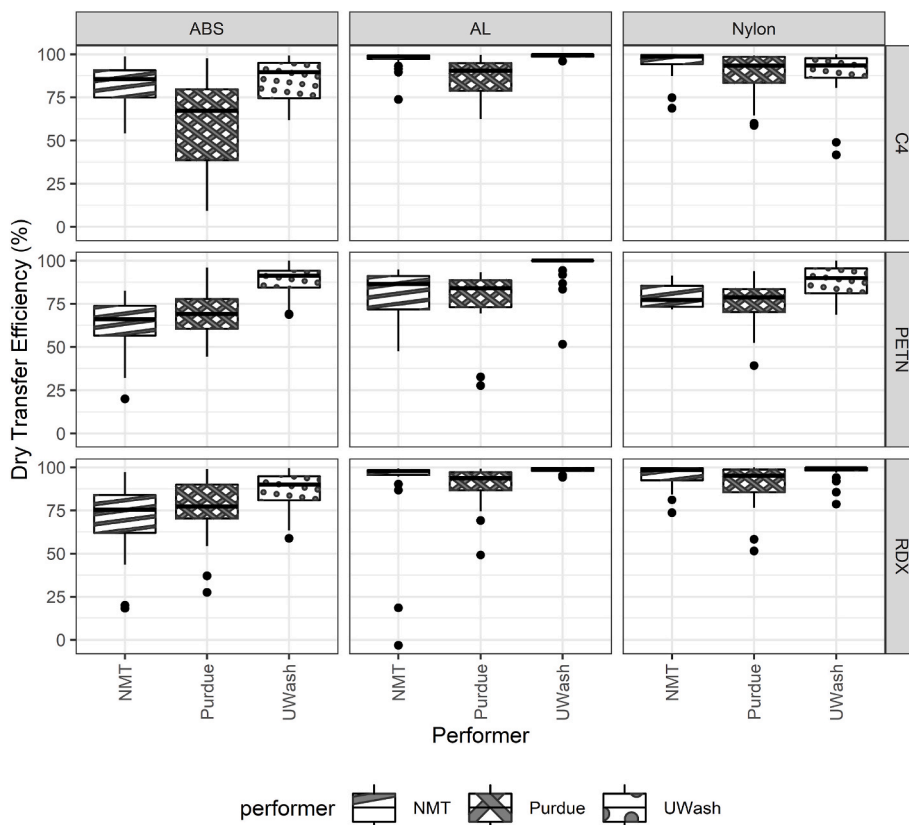

Fig. 8. Summary of the round-robin dry transfer results for C-4, PETN, and RDX residue onto ABS plastic, Aluminum, and Nylon surfaces. Generally, DTE >80%; however, significant variability was observed between the performers.
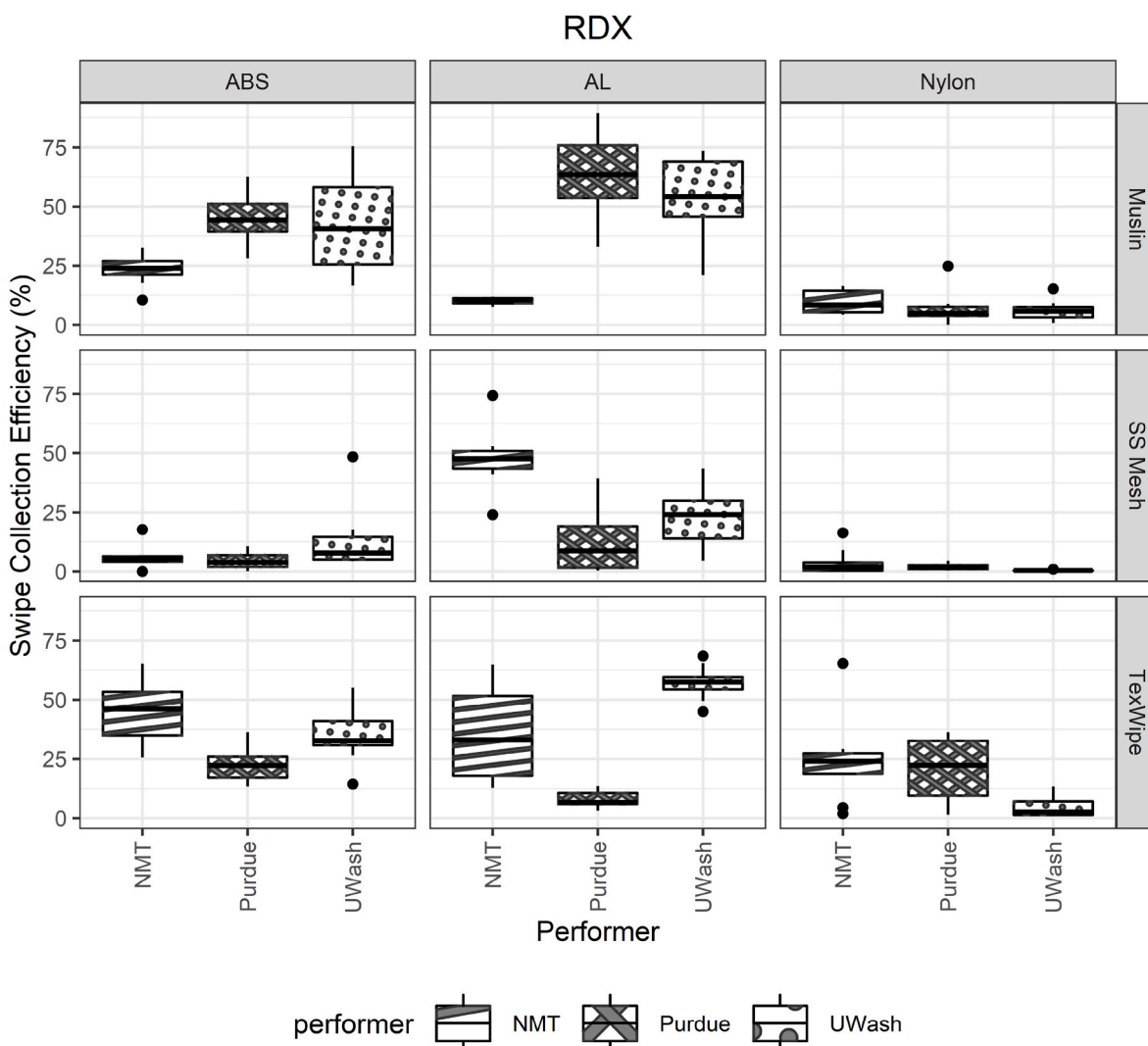
7

RDX

Fig. 9. Collection efficiency of RDX from ABS, AL, and NYL using three different traps. Generally, SS performed the worst, while the Texwipe cotton and MLN showed better performance across all the residues and surfaces.

3.2. Dry transfer efficiency

After the residues were transferred onto the surfaces via the dry transfer, the spent PTFE strips were extracted, and the amount of explosive residue was quantified to assess the DTE. High DTEs for all three explosives were noted on each surface, as shown in Fig. 8 as a boxplot. The lowest DTE across all residues was for ABS plastic, with an average value of 79%. Out of the three tested explosives on the ABS plastic, PETN had the worst DTE with NMT, PUR, and UW reporting values of 62.2%, 69.9%, and 88.5%, respectively, while RDX had the highest and the most consistent deposition with NMT, PUR, and UW reporting values of 72.7%, 77.1%, and 87.0%, respectively. Deposition onto NYL and AL for all analytes and performers was very consistent, with DTE values ranging from 75 to 99%, and no significant trends observed. It is likely that both NYL and AL are sufficiently rough (RMS and peak-peak height) to allow a large number of contacts between residue on the dry transfer strip and asperities on each test surface, resulting in the residues being efficiently transferred to the test surface. ABS's smoother surface reduces the number of possible contact points between the residue and peaks or valleys on the surface.

3.3. Trap collection efficiency

Following the dry transfer, the residue was collected on the traps using the procedure described above in Section 2.4. In no cases was explosive residue detected on the foil placed under the trap; thus, in the calculation of CE%, sample losses through the trap have been ignored. Fig. 9 shows the CE% of RDX from the three different surfaces. The SS trap was the least effective trap, while the Texwipe cotton and MLN offered similar performance, on average. It is believed that the rigidity of the SS mesh prevented it from deforming into the features on the surfaces; therefore, limiting its ability to collect samples from the surfaces at tested loads. The ABS had the lowest RMS roughness of the surfaces studied, and the NYL was the roughest, as shown in Table 4. The CE% was worst from the NYL, likely because there were large open spaces in the NYL where residue could be deposited during the dry transfer, limiting availability for harvest. While the AL was rougher than the ABS, it was not substantially rougher. It appears that the less uniform roughness on the ABS was more effective at hiding the residue from the trap than the AL, leading to the lower sampling efficiency from the ABS than from the AL.

Analysis of C-4 (Fig. 10) and PETN (Fig. 11) follows the RDX trends. The collection was slightly more effective from the AL than from the ABS and was the lowest for the NYL. Significant variability in the collection was observed across all residues, surfaces, performers, and traps. The apparent differences in CE% as a function of residue composition were also observed. The removal efficiency for C-4 was the lowest, while for RDX, it was the highest. This observed difference is confirmed by
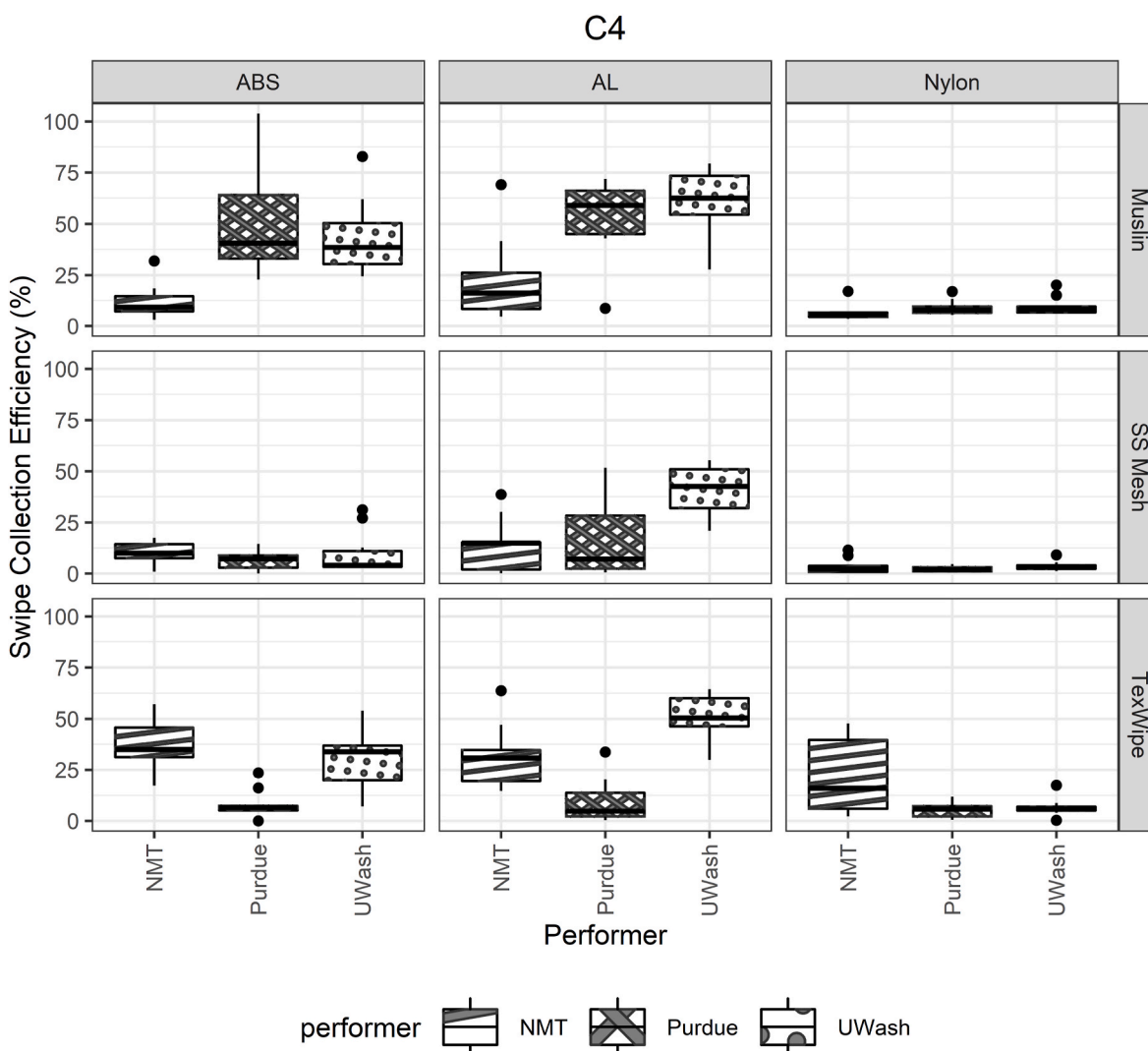
Fig. 10. Collection efficiency of C-4 from ABS, AL, and NYL for three different traps. SS generally performed the worst across all surfaces, while the Texwipe cotton and MLN performed comparably.

previously conducted atomic force microscopy (AFM) studies demonstrating that C-4 has the highest and RDX the lowest adhesion force to most surfaces among the three explosives being studied [6,18].

3.4. Collection efficiency from various surfaces

To determine the effect of the surface on which the residues were deposited on the collection efficiency, the following correlations were attempted:

1) For each residue, the CE of all traps was averaged over all performers and plotted as a function of either the RMS roughness of each surface type or the average peak – peak height of each surface type. No consistent trend was observed in either case.
2) For each residue, the CE of all traps was averaged over all performers and plotted as a function of either the standard deviation of the RMS roughness of each surface type or the standard deviation of the peak – peak height of each surface type. The standard deviation in the RMS roughness or the peak – peak height are both descriptors of the extent to which one will encounter unusually sized peaks or valleys on the surface. The results are shown in Fig. 12 and Fig. 13.

The standard deviation in the RMS roughness and the standard deviation in the peak-peak height are two related indicators of the

likelihood of encountering a randomly non-uniform feature on the contaminated surfaces. Such random non-uniform features provide locations where residue can 'hide' from the swabs, or where residue that is being removed from the surface can be scraped from the trap and left behind on the surface.

4. Conclusions

In this work, we have (i) evaluated a reproducible method for evaluation of collection efficiencies of trace explosive deposits by contact sampling, (ii) compared the CE% of different traps on model surfaces, and (iii) assessed the reproducibility of contact sampling in a multi-institutional study. We report the results of a round-robin test evaluating dry transfer and collection efficiencies of three explosive residues (RDX, C-4, and PETN) using three traps (Texwipe cotton, MLN, and SS) from three representative surfaces (ABS, AL, and NYL). This multi-institutional study assesses user bias and uncertainties related to the sample preparation, collection, and analysis. Though the testing parameters have been standardized among the participants and the analysis was performed by a third-party laboratory, high variability in DTE and CE% was observed for all performers, residues, traps, and surfaces. This has to be taken into account for the development of protocols and recommendations for real-world operations. Specifically, the trap's ability to access features on the surface and accommodate residue within
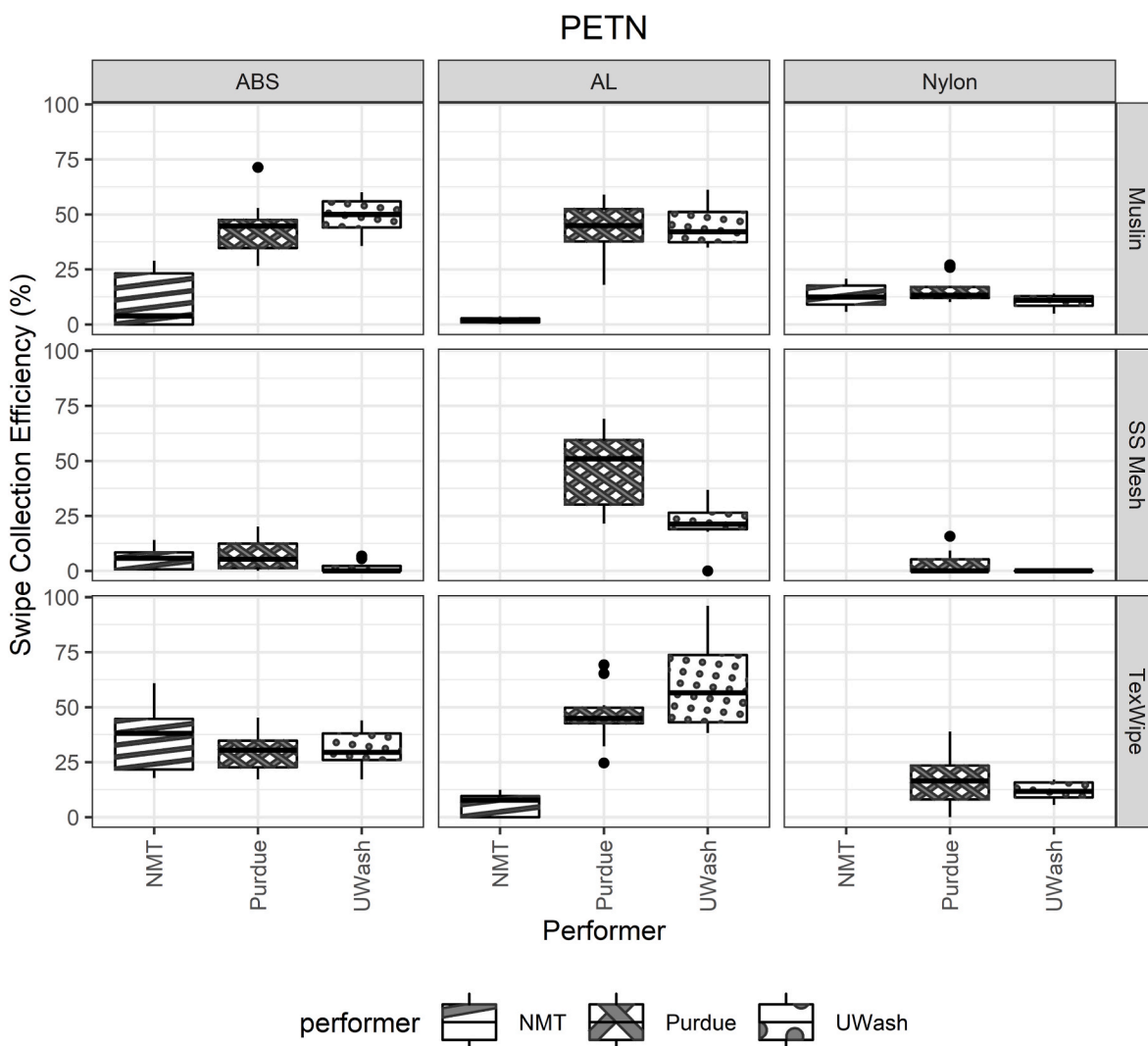
Fig. 11. Collection efficiency of PETN from ABS, AL, and NYL substrates for three different traps. Some data is missing due to sample handling errors. SS generally performed the worst across all surfaces, while the Texwipe cotton and MLN performed comparably.
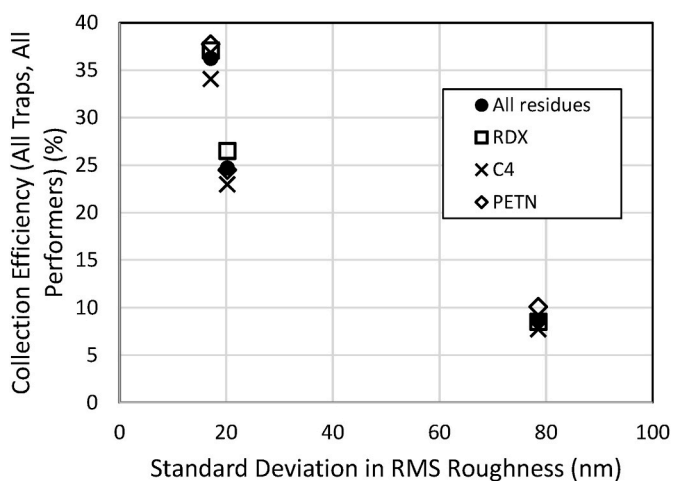

Fig. 12. Average CE (for all traps, all performers) of each threat, as a function of the standard deviation of the roughness of the surfaces. As the standard deviation of the roughness increases, the likelihood of encountering an unusually sized peak or valley on the surface is increased, and the CE decreases. The non-uniform features on the surface provide locations where residues can 'hide' from the collection trap.

the trap structure plays a substantial role in the sampling efficiency. Additional investigation into the environmental effects and sampling scenarios for model traps and surfaces, as well as the applied studies related to the more specific scenarios, are needed. This work establishes a generalized framework for evaluating contact sampling efficacy for trace explosives residues and other applications. This an initial study aimed to evaluate the generalized methods, a more comprehensive study of sampling efficiencies is in progress.

Author contributions

Igor V. Novosselov: Conceptualization, Methodology, Resources, Writing – original draft, Writing – review & editing, Visualization, Supervision, Caralyn A. Coultas-McKenney: Investigation, Data curation, Leonid Miroshnik: Investigation, Data curation, Kalyan Kottapalli: Investigation, Data curation, Writing – original draft, Visualization, Byron Ockerman: Investigation, Data curation, Tara E. Manley: Validation, Formal analysis, Investigation, Data curation, Myles W. Gardner: Validation, Formal analysis, Investigation, Data curation, Richard Lareau: Conceptualization, Methodology, Resources, John Brady: Writing – review & editing, Visualization, Melissa Sweat: Investigation, Data curation, Alan R. Smith: Conceptualization, Methodology, Validation, Formal analysis, Resources, Data curation, Writing – review & editing, Visualization, Supervision, Michael J. Hargather: Conceptualization,
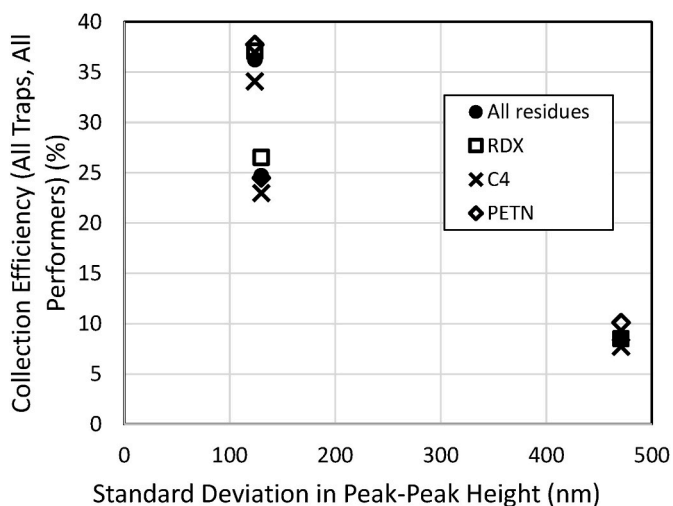
Fig. 13. Average CE (for all traps, all performers) of each threat, as a function of the standard deviation of the peak-peak height of the surfaces. As the standard deviation of the peak-peak height increases, the likelihood of encountering an unusually sized peak or valley on the surface is increased, and the CE decreases. The non-uniform features on the surface provide locations where residues can 'hide' from the collection trap.

Methodology, Resources, Writing – review & editing, Supervision, Stephen Beaudoin: Conceptualization, Methodology, Resources, Data curation, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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