TECHNICAL NOTE:

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A Recovery Method for Airborne Gunshot Residue Retained in Human Nasal Mucus

REFERENCE: Schwartz, R. H. and Zona, C. A., "**A Recovery Method for Airborne Gunshot Residue Retained in Human Nasal Mucus,**" *Journal of Forensic Science,* Vol. 40, No. 4, July 1995, pp. 659–661.

ABSTRACT: Each living person is equipped with their own personal particle filter, a nose. The human nose is capable of filtering and trapping airborne gunshot residue (AGSR) from discharged firearms. An extraction/concentration technique has been developed to recover the AGSR retained in human nasal mucus. The technique has successfully recovered abundant AGSR from 48 hours post-firing sample collection times. The AGSR particles were characterized by a JEOL™ 6400 scanning electron microscope coupled with a Noran™ Voyager energy dispersive X-ray spectrometer.

KEYWORDS: forensic science, gunshot residue, nasal mucus

The first documented criminal case of trace evidence being recovered from human nasal mucus dates back to 1904. Dr. Georg Popp identified particles trapped in the nasal mucus deposited on a handkerchief found near a crime scene. The particles in the mucus were linked back to a suspect, who after learning of this evidence, subsequently confessed to the crime [*1*]. Ninety years later we are returning to the human nose to examine inhaled AGSR particles trapped in human nasal mucus.

The literature states that after a person discharges a firearm, particles containing varying mixtures of Lead (Pb), Barium (Ba), and Antimony (Sb), or "classic" gunshot residue (GSR), maybe deposited onto the shooter [*2*]. Externally deposited particles are collected by one of several techniques [*3*]. The analytical instrumentation currently used to analyze the collected particles include; neutron activation analysis (NAA), flameless atomic absorption spectroscopy (FAAS), and scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS). The latter is potentially superior because it is a nondestructive technique that characterizes individual GSR particles both morphologically and elementally [*4,5*].

The externally deposited particles which are subsequently collected are known to be shed from the shooter as a function of time, therefore all current analytical techniques suggest short post firing collection times in most situations [*5*]. Conversely, particles of AGSR suspended in the air, are inhaled and trapped in the nasal mucus of the shooter and remain until purged, allowing for a much longer post firing sample collection times.

Received for publication 17 June 1994; revised manuscript received 29 July and 29 Sept. 1994; accepted for publication 22 Oct. 1994

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Presented at the 46th Annual Meeting of the American Academy of Forensic Sciences, San Antonio, Texas, 17 Feb. 1994.

Materials and Methods

The materials used in this study included: a suitable collection substrate, Corning™ 30 mL Vycor™ crucibles and lids, a Thermolyne™ 1400 muffle furnace, particle-free distilled water, 47 mm diameter stainless steel support vacuum filtration apparatus, 25 mm diameter sintered glass frit support vacuum filtration apparatus, 47 mm diameter, 0.3 μm pore size, mixed cellulose ester (MCE) membrane filters, 25 mm diameter, 0.8 μm and 0.4 μm pore size polycarbonate (PC) membrane filters, two 1000 mL sidearm vacuum filtration flasks, water aspirator, a table-top low power ultrasonic bath, fine forceps, and a JEOL™ 6400 SEM coupled to a Noran™ Voyager EDS. An SEM accelerating voltage of 25 kV was used and the analyzer employed was a lithium-drifted silicon crystal X-ray detector.

In this study, a shooter was exposed to AGSR from a single round of 38 Special caliber, nylon jacketed 158 grain Federal Register ammunition. The shooter was exposed to the AGSR for one minute under "normal" breathing conditions. The shooter then exited the room and washed his/her hands and face. The room was closed and evacuated, using exhaust fans vented to the roof outside to ensure a complete air exchange.

The air quality of the room was monitored for AGSR before, during, and after shooting, using standard air sampling techniques [*6–8*]. The air samples were collected on a 25 mm diameter (0.45 μm pore size) MCE membrane filter, housed in a 3 piece cassette, with a 50 mm electrically conductive extension cowl. The air samples consisted of drawing a known volume of air (1800 L), across a .45 μm pore size MCE membrane filter, during a recorded time interval.

A quarter of the filter membrane was removed with a clean scalpel and a carbon extraction replica of the filter was prepared for analysis by transmission electron microscopy (TEM) at 20,000X magnification. The room atmosphere did not contain any detectable AGSR by this method prior to shooting.

After a recorded post-firing time the nasal mucus sample is collected onto a 5″ × 5″ piece of substrate, by normal nose blowing. The evaluation of suitable collection substrates will be covered

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below. The used collection substrate is placed into a scrupulously clean 500 mL beaker and filled with enough particle free water, at 50–60°C to sufficiently submerge the collection substrate (200 mL). Immediately after the sample and collection substrate are immersed, the mucus will begin to delaminate from the collection substrate. Stirring the sample with a clean glass rod helps in loosening the nasal mucus and can be used to squeegee off any remaining mucus into the beaker.

The mucus and water are filtered onto a 47 mm diameter, 0.3 μm pore size, MCE membrane filter using a 47 mm diameter, stainless steel support screen vacuum filtration apparatus. Upon completion of filtration the 47 mm diameter, 0.3 μm pore size, MCE membrane filter is removed from the support screen and placed into a Vycor™ 30 mL crucible. The crucible is covered and heated in a muffle furnace at 550°C. The sample remains in the muffle furnace until a small amount of grayish-white ash is observed at the bottom of the crucible. Heating the sample to white ash can take from two to five hours, depending on the mucus quantity and moisture percentage.

The crucible is allowed to cool, filled to capacity with particle-free distilled water, and placed in an ultrasonic bath. Ultrasonication breaks up any ash aggregates and resuspends the particulate matter.

The contents of the crucible are then filtered onto a 25 mm diameter, 0.8 μm pore size, PC membrane filter using a 25 mm diameter, sintered glass-frit support vacuum filtration apparatus. The crucible should be thoroughly rinsed with particle free water until 100–125 mL of water has been collected in the sidearm vacuum flask. Wet PC filters can be taped down along two edges with Scotch™ or similar cellophane tape, onto a clean microscope slide and allowed to dry inside a covered petri dish. After the filters are completely dry they can be prepared for SEM/EDS analysis. Polycarbonate filters were used in the second filtration because they require very minimal preparation for analysis by SEM/EDS.

Discussion

The desirable characteristics for a collection substrate are: a suitable low temperature (under 550°C) ash, absence of delustering agents, ability to resist dissolution in water, and low cost. All collection substrates were purchased at local fabric stores.

The ashes of several substrates including; facial tissue, cotton, wool, silk, wool/polyester blend, polyester, acetate, and viscose rayon fabrics were evaluated for their particle content from ash and overall ash properties. The reason for such an evaluation was to determine if the sample and substrate could be directly ashed, thus eliminating the water extraction step and to determine the cleanest substrate available.

Substrate samples large enough to adequately collect a sample (5″ × 5″) were heated in a Vycor crucible in a muffle furnace at 550°C to white ash. All ashes were suspended in particle-free water and filtered onto a 25 mm diameter, 0.45 μm pore size, MCE membrane filter. A carbon extraction replica was prepared from a portion of the filters and examined by TEM at magnifications of 10,000 to 20,000 X. The particles were identified by morphology and EDS [7].

There were many problems associated with the direct ashing method and varied depending on the material. In general, all substrates required much longer heating times in order to obtain a white ash. Secondly, the amount of ash generated is substantial, increasing filtering times and potentially obscuring AGSR during analysis. The AGSR samples prepared by the direct ashing method consistently recovered fewer particles upon analysis by SEM/EDS. It is believed that the same amount of AGSR particles were recovered by this method, but were obscured by the ash during SEM/EDS analysis.

This study of the ash also helped to determine the "cleanest" substrate. By using the cleanest substrate possible, the risk of filtering extraneous particles with the sample is minimized. The collection substrates containing the least amount of particles from ash were said to be the "cleanest."

Facial tissues, and paper products in general, produce very high, often obscuring calcium backgrounds in the EDS spectrum. The natural fabric products (cotton, wool, and silk) all contained large amounts of inorganic particles and general filth that also resulted in high backgrounds. The nondelustered synthetic fabric substrates (cellulose acetate, polyester, and viscose rayon) exhibited varying ashing properties and were generally "cleaner" than the natural products. The cleanest fabric collection substrate was viscose rayon. Polymer films (household plastic wraps) were also evaluated as collection substrates. They are generally particle-free and waterproof, however, did not adequately ash at the low temperature employed.

Initially there was concern over using an ashing temperature greater than the melting point of Pb (327°C) and the melting point eutectic of Pb/Sb (251°C) to ash the filtered sample. Undoubtly the AGSR particles are melting when exposed to temperatures above the melting point of Pb. However, the particles identified as GSR maintained a spherical morphology. One explanation is that the lattice formed from the white ash of the MCE membrane filter and mucus, support the AGSR spheres and allow them to cool undisturbed. Other viable explanations have been made but are beyond the scope of this work. Currently research is being carried out on the use of plasma ashing as a substitute for muffle furnace ashing.

The nasal mucus, as mentioned earlier, is collected by normal nose blowing. From a practical stand point the woven fabrics are easier to use because they can be used like an ordinary handkerchief. The collection of nasal mucus onto household plastic wrap is problematical because of its lack of absorbency, especially with larger samples.

Sample collection onto fabrics and polymer films was favored over swabbing the nasal cavity. Swabbing proved to be too invasive, causing overall discomfort and occasional nasal bleeding. Swabbing also did not collect as large a quantity of mucus as the blowing method. It was for these reasons that the swabbing approach to nasal mucus collection was discarded for living individuals.

Results

All samples were mailed from Chicago, IL to Norcross, GA and submitted as a blind study. The sample population consisted of: AGSR samples, nasal mucus blank samples, collection substrate blank samples, laboratory blank samples and AGSR persistence samples.

The AGSR samples consisted of drawing two liters of air containing AGSR, across a 25 mm, 0.45 μm pore size, MCE membrane filter, using a Gillian® low volume air pump. The low volume AGSR air samples were used to make a conservative determination of the amount of AGSR a person could potentially inhale during a one minute time interval. The average person inhales between 6–16 liters of air per minute under "normal" breathing conditions.

TABLE 1—*Recovered inhaled gunshot residue.*

Sample	Total Area Searched in mm^2	Total Recovered Particles	Total Recovered GSR Particles	Normalized Recovered GSR Particles/mm^2	PFCT[a]	Collection Substrate
1	3.5	976	16	4.6	12 Hours	Rayon
2	4.5	920	75	16.7	15 Hours	Rayon
3	5.76	275	233	40.4	48 Hours	Rayon
4	4.6	737	49	10.6	48 Hours	Rayon
5	2.0	902	11	5.5	48 Hours	Saran
6	3.4	854	542	159.4	48 Hours	Saran

[a]Post firing collection time.

The two liter air samples were examined by TEM and contained abundant recovered AGSR particles.

The nasal mucus blank samples consisted of subjects that frequented urban or rural settings. The subjects were asked if they had recently discharged a firearm and their current occupation. If the individual had been in contact with a firearm this was noted. Collection substrate blanks were prepared and submitted for analysis by SEM/EDS throughout the study to ensure that the substrates had not become contaminated. Laboratory blanks consisted of 25 mm, 0.8 μm and 0.45 μm filter samples from the particle-free water, crucibles and glassware to monitor the overall cleanliness of the laboratory equipment.

The persistence samples ranged in post firing collection times from 12 to 48 hours. The preliminary persistence findings show over 500 AGSR particles were recovered from a nasal mucus sample of a shooter, after a 48-hour post firing time (see Table 1). The problem with determining the maximum post firing collection time (greater than 48 hours) is with the subject. It is very difficult not to blow ones nose for longer than a 48-hour period.

The total number of particles and AGSR particles recovered will vary between individuals. Each human nose is anatomically different and will filter airborne particulate with different degrees of efficiency, depending on the subjects health and occupation, the time of year, and geographic location.

Conclusions

This study is by no means exhaustive in terms of persistence. The primary goal of this study was to develop a recovery method for AGSR retained in the nasal mucus of a shooter. The substantial number of AGSR particles recovered from the nasal cavity and its persistence were other significant findings of this study. Currently, AGSR research is being carried out focusing on the persistence of AGSR in the human nose using different caliber weapons, ammunition, and post-firing collection times.

Acknowledgments

The authors would like to thank Dr. and Mrs. Donald Schwartz for their financial support. A very special thanks to Dr. and Mrs. Walter McCrone and Dr. David Stoney for approving this research as an in-house project. This work could not have been completed without the expertise of Richard S. Brown of MVA, Norcross, GA. Finally, this paper is dedicated to Donald P. McManigal, J.D., who was taken from us far too early.

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