

TECHNICAL NOTE

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The Application of Prefiltration to the Analysis of Acid-Degraded Gunshot Residue Swabs

The collection of gunshot residues (GSR) from a suspect's hands is a routine forensic technique widely utilized by police investigators. Presently the most widely used method for the collection and analysis of GSR samples is by swabbing the hands with dilute nitric-acid-moistened cotton swabs and determining the barium and antimony levels by flameless atomic absorption spectrophotometry (FAAS). These techniques of collection and analysis have acquired wide acceptance because of their low cost, uncomplicated procedures, and overall reliability when compared with alternate methods.

Although the routine analysis of GSR swabs by FAAS is straightforward with clean swabs, severe problems are encountered with swabs that are acid-degraded or are contaminated by grease, dirt, or blood. In these situations the analysis is complicated by increased instrumental backgrounds, decreased leaching effectiveness, and "memory" between subsequently analyzed samples. The most commonly encountered problem is swabs that have degraded because of attack of the nitric acid on the cotton tip of the swabs. This problem is most severe when the swabs dry after collection but before analysis. The cotton tip of the acid-degraded swabs appears dry and powdery in texture and will disintegrate during the leaching stage of the analysis, which places the cotton fibers in suspension in the leachate (Fig. 1). This degradation process does not appear to occur in cotton swabs that have not dried out in storage. A reasonable explanation is that during drying water evaporates from the acid on the swabs, thus concentrating the acid. The more concentrated acid will then attack the cotton tips, causing degradation. The analysis of degraded swabs for antimony by FAAS is typified by high instrumental backgrounds, memory between subsequently analyzed samples, and objectionable signal to background ratios. Low-temperature oxygen plasma ashing has been used by Kinard and Midkiff [1] to alleviate these problems with swabs contaminated by blood, grease, or dirt. The present study was initiated to determine if a simple prefiltration technique could minimize these difficulties when acid-degraded swabs are analyzed.

Instrumentation and Equipment

The filtration apparatus consists of a Nuclepore polycarbonate membrane filter disk (13 mm diameter, 3.0 μm pore size, #N300CPR01300) held in a Millipore Swinny filter

Received for publication 23 March 1978; revised manuscript received 10 June 1978; accepted for publication 14 June 1978.

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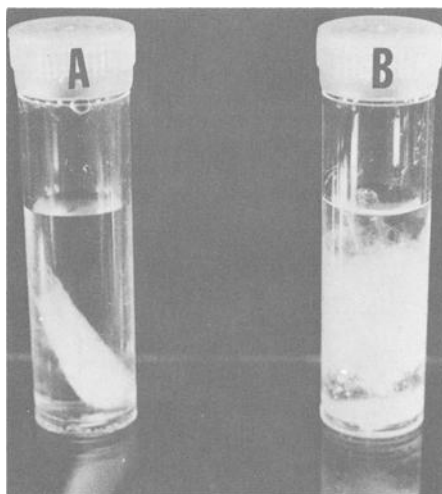


FIG. 1—Typical appearance of (A) undegraded GSR swab and (B) acid-degraded GSR swab.

holder (#XX30-012-00) and a standard 5-ml plastic syringe. The items were rinsed in 1M nitric acid to remove any residual antimony.

A Jarrell-Ash Model 810 dual-channel double-beam atomic absorption spectrophotometer, equipped with a tantalum strip atomizer, was used for the antimony determinations. Instrument operating parameters, preparation of analytical curves, and sample handling procedures have been described by Goleb and Midkiff [2].

Experimental Procedure

Single-ended plastic-shafted cotton swabs were spiked with 5.0 μg of antimony in 1M nitric acid. The spiked swabs were stored in individual antimony-free polystyrene vials and allowed to degrade for two weeks at room temperature. At the end of this period the cotton was observed to be loose and powdery in texture and resembled the degraded swabs common to case samples.

New single-ended plastic-shafted cotton swabs spiked with 0.5 μg of antimony were used as controls and similar unspiked swabs were used as blanks.

To leach the antimony from the swabs, 1 ml of 1M nitric acid was added to each vial; the vials were then capped and agitated in an oscillatory shaker for 15 min. A 10- μl aliquot of the leachate was pipetted on the tantalum strip of the spectrophotometer. Absorbance tracings were obtained by FAAS at the antimony analytical line at 217.6 nm while background was monitored at the related antimony background correction line at 217.9 nm.

After the absorbance tracings of the degraded swabs were obtained the effectiveness of the filtration process was tested by filtering the remaining leachate from the degraded swabs, containing cotton fibers, through a 3.0- μm pore size Nuclepore filter membrane held in a 13-mm diameter Millipore Swinny filter holder, with a standard 5-ml plastic syringe. The filtrate was collected in an antimony-free polystyrene vial and the absorbance tracings were obtained.

Results and Discussion

The results of the prefiltration technique are illustrated in the absorbance tracings of

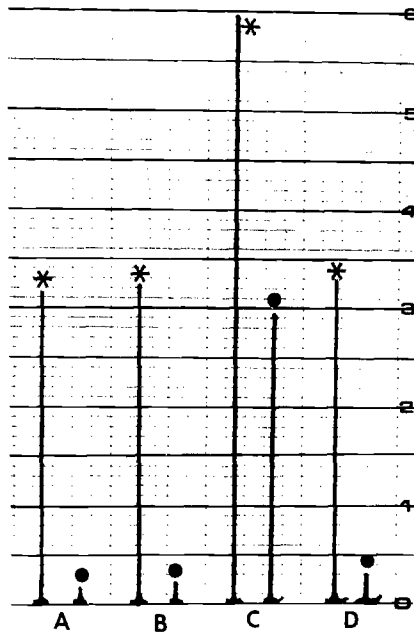


FIG. 2.—Absorbance tracings of analytical signal (*) and background correction signal (●) for (A) 0.5 μg of antimony per millilitre of nitric acid standard solution, (B) undegraded swab spiked with 0.5 μg antimony, (C) degraded swab spiked with 0.5 μg antimony, and (D) filtrate from degraded swab spiked with 0.5 μg antimony.

Fig. 2. An unfavorable signal to background ratio of approximately 2:1 was observed with the acid-degraded swabs. A much more favorable signal to background ratio, of approximately 10:1, was obtained where the leachate from the acid-degraded swabs was prefiltered before analysis. This signal to background ratio approximates that obtained in the leachate from undegraded swabs.

The high instrumental backgrounds observed in the analysis of the unfiltered leachate from the acid-degraded swabs appear to originate from a small amount of cotton fibers suspended in the solution and transferred to the tantalum strip of the atomizer. It appears that these cotton fibers only char, and do not completely ash, during the ashing cycle when the temperature of the tantalum strip is maintained at 450°C for 30 s. During the atomization cycle, when the tantalum strip reaches a temperature of 2500°C, the remaining unashed cotton fibers are ashed and the resulting smoke causes scattering of both the analytical and background beams.

Traces of the partially charred cotton fibers also occasionally remain on the tantalum strip of the atomizer. These fibers apparently cause the "memory" of approximately 0.02 to 0.04 μg antimony between subsequently analyzed samples. These charred fibers may remain on the strip after repeated post-firing operations, which normally clean the strip of extraneous materials. Another consequence of these adhering fibers is that they apparently cause localized temperature differences on the strip during atomization, greatly reducing strip life.

While the technique of prefiltering the leachate from acid-degraded GSR swabs is a viable method for reducing background scattering effects and memory between samples, it does increase sample handling procedures and lengthen analysis time. Despite these minor inconveniences, prefiltration of acid-degraded cotton swabs is an attractive and inexpensive method of improving analytical precision in the analysis of GSR swabs.

References

- [1] Kinard, W. D. and Midkiff, C. R., Jr., "The Application of Oxygen Plasma Ashing to Gunshot Residue Analysis," *Journal of Forensic Sciences*, Vol. 23, No. 2, April 1978, pp. 368-374.
- [2] Goleb, J. A. and Midkiff, C. R., Jr., "The Determination of Barium and Antimony in Gunshot Residue by Flameless Atomic Absorption Spectroscopy Using a Tantalum Strip Atomizer," *Applied Spectroscopy*, Vol. 29, No. 1, Jan./Feb. 1975, pp. 44-48.

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