Ronald L. Singer,[1] M.S.; Dusty Davis,[2] B.S.; and Max M. Houck,[3] M.A.

A Survey of Gunshot Residue Analysis Methods

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ABSTRACT: A survey was sent to 80 forensic laboratories in 44 States and two Canadian Provinces concerning methodology in analyzing gunshot residue (GSR) and interpreting the results. Of the 80 surveys, 50 (63%) were returned completed. Questions included standard procedures, collection methods, thresholding problems and specificity of data. These results are compared to a previous survey reported in 1990. Implications for the interpretation and future study of these methods are discussed.

KEYWORDS: forensic science, criminalistics, gunshot residue, scanning electron microscopy, energy-dispersive spectrometry, X-ray emission spectrometry, electron probe microanalysis, atomic absorption spectrophotometry, methodology

Gunshot residue (GSR) has been dealt with as analytical evidence in suicides, homicides and other firearms related incidents for many years and has received a good deal of attention in the literature. The majority of these papers, however, have dealt with GSR as either a case-study or technical report (see 1, for example) or an analytical technique (2,3) Two papers that have addressed the methods of analyzing GSR are those of Germani (4) and DeGaetano and Siegel (5). Germani's paper offers an approach to standardizing parameters for scanning electron microscopy/energy-dispersive spectrometry (SEM/EDS) to increase the accuracy of the technique while achieving optimum time-per-analysis. The paper by DeGaetano and Siegel, however, reports on a survey of current GSR analysis methods in forensic laboratories, focusing on the most common analytical methods, atomic absorption (AA) and SEM/EDS. The research for their paper was performed in 1988 and reported on in 1990.

To follow-up on any changes in GSR methodology and to assist in the standardization of the technique among forensic scientists, a new survey was conducted using, with their permission, a subset of DeGaetano and Siegel's original sample. The questions were designed to address specific problems in the analysis of GSR and the interpretation of GSR results. Participants were asked in they performed GSR analyses themselves, sent the samples to an outside agency or if no GSR analyses were requested. If the laboratory performed analyses or sent out samples, they were asked to indicate the methods used. If no GSR analyses were requested, the survey was then to be returned. Of the methods on the list, atomic absorption, inductively-coupled plasma emission (ICP), neutron activation analysis (NAA) and scanning electron microscopy/energy-dispersive spectrometry, the laboratories were to answer additional questions specific to AA and SEM/EDS as completely as possible if they performed or requested analyses by those methods. The only other question asked of all laboratories was if GSR kits from suicides were routinely analyzed or submitted.

[1]Chief Criminalist, Tarrant County Medical Examiner's Criminalistics Laboratory, Fort Worth, TX and Institute of Forensic Medicine, Texas College of Osteopathic Medicine, Fort Worth, TX.

[2]Forensic Chemist, Retired, Tarrant County Medical Examiner's Criminalistics Laboratory, Fort Worth, TX.

[3]Examiner, F.B.I. Laboratory, 10th and Pennsylvania Avenues, N.W., Washington, D.C.; Formerly, Trace Analyst, Tarrant County Medical Examiner's Criminalistics Laboratory, Fort Worth, TX.

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Methods

A four-page questionnaire (see Appendix A for a copy of the survey) was sent to 80 forensic science laboratories in 44 U.S. States and two Canadian Provinces; participants had approximately two months to fill out the survey and were given a self-addressed, stamped envelope to facilitate its return. Laboratories were chosen on the basis of a list generated for DeGaetano and Siegel's survey performed in 1988; only those members of the original list who returned the survey on the first pass were included in the present survey. It was hoped that this judgement sampling would elicit a higher rate of return. Copies of the results were made available to any participating laboratory upon request.

Results and Discussion

Of the 80 surveys sent, 50 (63%) were returned by the requested date. Who performs the analyses of GSR and a comparison of these findings with those of DeGaetano and Siegel can be seen in Table 1. Fifty-two percent of the laboratories analyze GSR themselves, 35% send the samples to an outside agency and 13% do not request GSR analyses at all. As can be seen, GSR is analyzed more frequently than in the past and more labs are performing the analysis within their own laboratories.

Of the methods listed in the survey, the majority (44%) utilize the bulk method of AA for GSR analysis (Table 2); this shows only a slight decrease (−4%) over the value reported by DeGaetano and Siegel. Analysis by SEM/EDS, however, has shown a slight increase from 21% to 26% in the current study; the use of both

TABLE 1—*Changes in who performs analyses of GSR.*

	1990 Survey	1992 Survey	Change
In-House	43%	52%	+9%
Outside Agency	30%	35%	+5%
Don't Request GSR	27%	13%	−14%

TABLE 2—*Changes in analysis by instrumentation.*

Method	1990 Survey	1992 Survey	Change
Atomic Absorption Only	48%	44%	−4%
SEM/EDS Only	21%	26%	+5%
AA and SEM/EDS	13%	29%	+16%
NAA	1.6%	2%	+0.4%
ICP	4%	n/a	n/a

AA and SEM/EDS together has jumped from 13% to 29%. DeGaetano and Siegel noted in their study that SEM/EDS analyses were challenged less frequently in court than the results of bulk methods, and the increase in SEM/EDS use, by itself and with AA, seen in this study may be a result of that reliability. Well over half of the respondents routinely analyze or submit GSR kits from suicides (62%).

The percentage of the SEM stub surface area which is searched is crucial for statistical sampling purposes and the accuracy of any statements about the sample (see 6 and 7 for discussion). In the current study, over 72% of the laboratories analyzing GSR by SEM/EDS search more than 50% of the stub; some laboratories (18%) search as little as 10–20% of the stub's surface before confirming a negative result. The appropriate amount of search area on the stub has been discussed in the literature (3,6,7), but any discussion of search area begs the question: Are there sufficient GSR particles to consider this sample "positive?" Yet, many laboratories are unwilling or unsure about what constitutes a "sufficient" number of unique particles. Short of test firing comparative ammunition in the same type of weapon used in the crime, analysts cannot point to one or two references and produce the universal magic number for what constitutes a positive. Only two laboratories gave an answer to the question, "How many particles of GSR would be needed to indicate in a report that a person had been in the vicinity of a firearm being discharged or had discharged a firearm?"; one laboratory responded with "1" and the other answered, "2." The rest of the laboratories said that the criteria were "under advisement," "under consideration," "depended upon the types of particles found" and similar responses. In fact, the category "Interpretation in General" in DeGaetano and Siegel's survey is the most frequent reason that GSR results by SEM/EDS are challenged in court testimony (5).

If, as a matter of validated protocol, a "particle threshold" could be established, then the appropriate search area would be whatever area was searched until that number of particles was found; the

TABLE 3—*Ratings of particle elemental profiles for GSR definition by number of responses; blanks indicate no responses.*

	(GSR) 1	2	3	4	(Non-GSR) 5	Mode
PbSbBa	11					1
SiCaBaS	2	3	3		3	2,3,5
PbSb	1	7	4			2
PbBa	1	6	4			2
SbBa	4	5	1	3		2
Pb		1	6	3	1	3
Sb			5	4	1	3
Ba			5	1	2	3

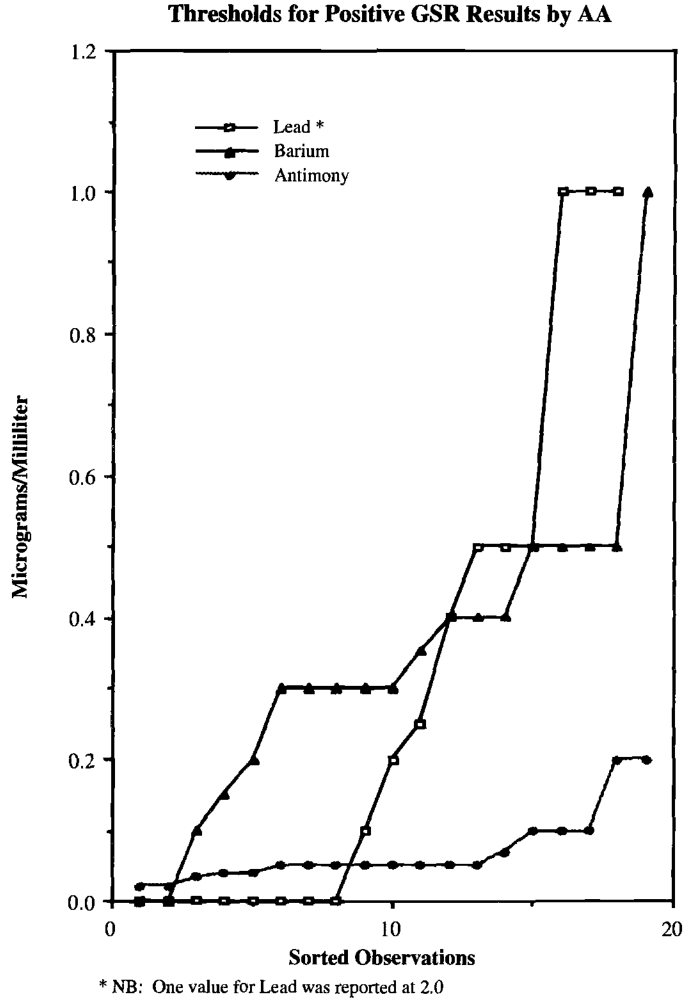

FIG. 1—*Thresholds for positive GSR results by AA.*

analyst or, if the system is automated, the software could then move to the next sample. This might take the form of, "if the number of (unique/indicative) particles is greater than *x*, then the sample is positive, if the number is below *x*, then it is indeterminate." A complete search of the stub surface would be required to confirm an indeterminate sample in any event to limit reporting a false negative, with the understanding that searching the entire stub will not always avoid a false negative. Currently, an analyst could only make such a statement about a particular firearm/ammunition combination after producing and analyzing several samples.

To collect GSR samples for SEM/EDS analysis, 72% use some sort of adhesive tape, which has been shown to be the most efficient method of collection (8); 21% use the glue-lift technique developed by Basu (9) and 7% use a concentration method (8,10). Since adhesive tape has been shown to be the most efficient method of GSR collection for SEM/EDS, an evaluation of the various types of adhesive tapes commercially available would help to optimize this collection method.

One of the goals of this survey was to determine which particle families are considered to be unique, indicative of, or unrelated to GSR. Wolten et al. (3) provided a taxonomy for classifying particles by some discussion concerning the specificity of some of those categories given current primer compositions has been published (11). Participants were asked to rate elemental profiles

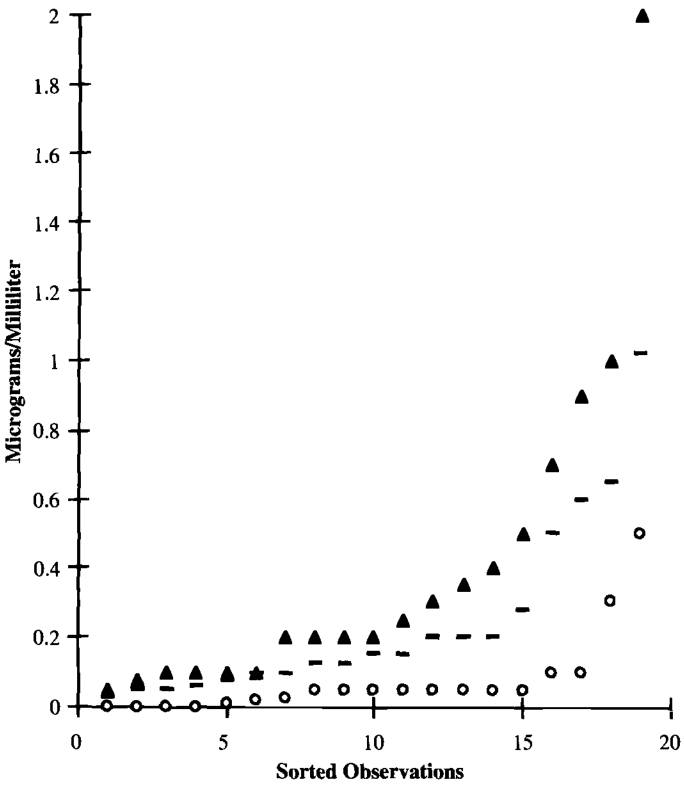

FIG. 2—*Sorted antimony calibration levels for AA.*

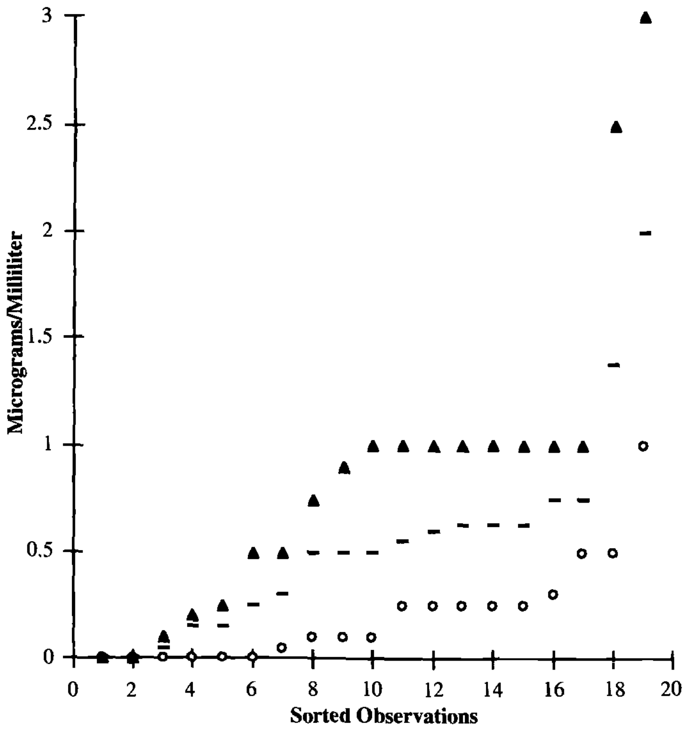

FIG. 3—*Sorted barium calibration levels for AA.*

of particles on a scale of 1 (unique to GSR) to 5 (non-GSR material); the results are shown in Table 3. The category most traditionally definitive to GSR, PbSbBa, is still considered to be the most specific; all other categories are considered indicative but not specific to GSR. The SbBa category may be an exception, since it rated higher as "unique" than any other category except PbSbBa. The SiCaBaS category is equivocally tri-modal with ratings of 2, 3, and 5.

Turning to the reporting of results, 55% of the respondents report unique and indicative particles, 36% report unique particles only and 9% report all categories of particles. In their survey, DeGaetano and Siegel had no difficulty with laboratories responding to the question of how many particles are needed to confirm the presence of GSR (5, p. 1091, Fig. 3), with 50% of their respondents requiring only one particle for confirmation and answers ranging from 1–10. The present study had only two laboratories which were willing to provide a concrete answer: One laboratory responded with "1" and the other answered "2." The number of particles found are not, however, reported by 83% of the laboratories in the current survey. While only one "three-component" (PbSbBa) particle is definitely GSR, any interpretation, including "GSR was present" with no discussion as to why only one particle might be present, could be misleading. Rates of deposition, transfer, contamination and loss of GSR are not well understood for particulates and this information is critical to any proposed interpretation. Simply stating that GSR was present in the sample can be misleading, much as testifying that automotive paint was found on the victim's clothing without accounting for which car it came from. On the other side of the argument, what makes one particle of GSR less significant than 20? The range of conclusions is the same (discharge, proximity, handling) and the number of particles found does not necessarily change the results. Some of the laboratories responding to this survey must be employing some sort of intuitive or discrete particle threshold, however, because they are actively analyzing GSR by SEM/EDS and reporting out conclusions, but, as this survey shows, they are not willing to report it.

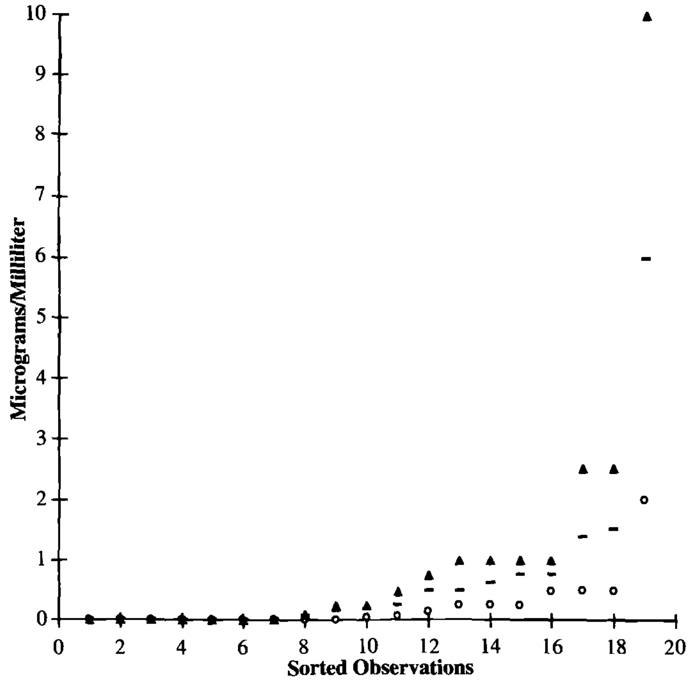

FIG. 4—*Sorted lead calibration levels for AA.*

The wording of reports is crucial to the accuracy and significance of the data in the courtroom. Given three levels of specificity of positive results, 80% of the respondents chose, "The sample is consistent with the suspect having discharged a firearm, having been in the vicinity of a firearm when it was discharged, or having handled an item with GSR on it," as the statement that most closely resembled the phrasing in their reports. Only 20% chose a more specific statement, "The sample is consistent with the suspect having discharged a firearm or having been in the vicinity of a firearm when it was discharged" and none chose the most declarative statement, "The sample is consistent with the suspect having discharged a firearm."

Additionally, 83% of the SEM/EDS systems in the laboratories polled are automated and 92% use the SEM/EDS system for trace analyses other than GSR. This is a strong statement against the common misconception among forensic laboratories that a SEM/EDS unit is necessarily dedicated to GSR analysis and serves no other purpose to a trace or chemical analysis section.

All of the laboratories that use AA for GSR analyses use Sb and/or Ba to test for the presence of GSR; only half use Pb in some combination with Sb or Ba. The most frequent response for elements that must exceed their threshold for a sample to be considered positive for GSR are, in order, Sb (22 responses), Ba (20 responses) and Pb (9 responses). Given these values, Pb is the element considered least specific to defining GSR; to put it another way, 63% of the laboratories responding do not even test for the presence of Pb in a GSR analysis. All three elements are screened for by 42% of the laboratories; the rest screen for one element only, either Sb (77%) or Ba (23%). As Pb is the most commonly encountered of these three elements in modern human society, it would seem that most laboratories consider it non-specific enough for GSR to not use its presence or absence as conditional for the presence of GSR.[4]

A surprising result of this survey is the range of values reported for threshold required for a positive result (Fig. 1) from 0 (not tested for) to 2.0 μg/mL for Pb, 0–1.0 μg/mL for Ba and 0–0.2 μg/mL for Sb. With the possible exception of Sb, no standardized threshold levels are currently in use in the forensic science community. The levels of instrumental calibration also vary considerably from laboratory to laboratory (Figs. 2–4). The descriptive statistics for these data are shown in Table 4; median values are reported as they tend to be more resistent to outliers.

A swab is included in the digestion of a calibration standard by 54% of the laboratories and a blank is analyzed for quality control by every laboratory in this survey. It would have been useful to have phrased the question to find out if the blank was a swab from the kit or just a reagent blank; if the answer would have been the latter, that would render this information less useful.

TABLE 4—*Descriptive Statistics for Positive Thresholds and Calibration Values (all values are in μg/mL).*

	Median Positive Threshold	Range	Median Minimum Calibration	Median Maximum Calibration
Lead	0.5	0–1.0	0.25	1.0
Barium	0.4	0–1.0	0.25	1.0
Antimony	0.05	0.02	0.05	0.20

[4]The abundance of Barium (390 p.p.m.), Lead (13 p.p.m.) and Antimony (0.2 p.p.m.) in the Earth's crust might indicate otherwise, but Lead is more commonly utilized in industry and production (12).

Summary

By surveying current practices in analyzing GSR, it is hoped that more consistent methodologies can be developed and more accurate results obtained. Although no single "correct" method exists, it is useful to examine what our peers employ as standards with an eye towards developing a consensus opinion on the minimum necessary requirements for an adequate analysis. This has been done for GSR collection and analysis by SEM/EDS (13) and should be considered for AA, especially given the range of values reported on in this survey. It is not envisioned that a "magic number" for what is a positive result will be generated but only that the way in which that result is achieved follows certain guidelines. Surveys such as the current paper should be performed occasionally so that we can all remain current with each other and increase the flow of information among crime laboratories.

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Address requests for reprints or additional information to
Ronald L. Singer
Tarrant County Medical Examiner's Criminalistics Laboratory
200 Feliks Gwozdz Place
Fort Worth, TX 76104-4919

ERRATUM

The following Appendix was inadvertently left out of "A Survey of Gunshot Residue Analysis Methods" by Singer et al. in the March 1996 issue of JOFS.

APPENDIX A

Survey Form

If your lab analyzes gunshot residue (GSR), please answer the questions for your laboratory; if you have an outside agency analyze your GSR samples for you, please answer the questions as completely as possible for the results that you receive from that laboratory.

1. Does your laboratory analyze GSR or do you have an outside agency analyze it for you?
   ____ We analyze GSR
   ____ Outside agency (Agency ____________________)
   ____ We do not request GSR analysis
2. Which of the following methods do you or the outside agency use?
   ____ AA
   ____ ICP
   ____ NAA
   ____ SEM/EDS
   ____ Other ______________
3. Do you analyze or submit GSR from suicide victims?
   YES NO

If your GSR is analyzed by SEM/EDS, answer Questions 4–17; if your GSR is analyzed by AA, answer Questions 18–26. If both methods are used, please answer all questions.

4. what percentage of the stub surface is searched (to the nearest 10%)? ____
5. Which collection method is used?
   ____ Adhesive Tape
   ____ Glue Life
   ____ Concentration
   ____ Other ______________
6. Please rate the following particle categories on their specificity to identifying GSE, with 1 being UNIQUE TO GSR and 5 being a NON-GSR MATERIAL.

PbSbBa	1	2	3	4	5
SiCaBaS	1	2	3	4	5
PbSb	1	2	3	4	5
PbBa	1	2	3	4	5
SbBa	1	2	3	4	5
Pb	1	2	3	4	5
Sb	1	2	3	4	5
Ba	1	2	3	4	5
Other:					
______	1	2	3	4	5

7. Which categories of particles are reported?
   ____ Unique GSR only
   ____ Unique GSR and indicative particles
   ____ All categories
8. How many particles of GSR would be needed to indicate in a report that a person had been in the vicinity of a firearm being discharged or had discharged a firearm? ______
9. Are the number of particles found reported?
   YES NO
10. Is GSR documented as being present in the sample by:
    ____ Written report
    ____ Written report and printout of the spectrum
    ____ Report, spectrum printout and a representative photo of a GSR particle
    ____ Report, spectrum printout and multiple photographs of GSR particles
11. Which of the following statements most closely resembles the phrasing used in your reports?
    ____ The sample is consistent with the target person having discharged a firearm, having been in the vicinity of firearm when it was discharged, or having handled an item with GSR on it.
    ____ The sample is consistent with the target person having discharged a firearm or having been in the vicinity of a firearm when it was discharged.
    ____ The sample is consistent with the target person having discharged a firearm.
12. What brand of SEM do you own? ________________
13. How old is it? ________ years
14. What brand of EDS system do you own? ______________
15. How old is it? ________ years
16. Is the SEM/EDS system automated? YES NO
17. Do you use your SEM/EDS system for trace analyses other than GSR? YES NO
18. Which elements do you test for with AA? (check all that apply)
    ____ Pb
    ____ Ba
    ____ Sb
19. Which elements must exceed their threshold for a sample to be considered positive for GSR? (Check all that apply)
    ____ Pb
    ____ Ba
    ____ Sb
20. What are the thresholds required for a positive result for each of the following elements?
    Pb ____ μg/ml
    Ba ____ μg/ml
    Sb ____ μg/ml
21. What levels of calibration standards are used?
    Pb ____ μg/ml
    Ba ____ μg/ml
    Sb ____ μg/ml
22. Is a swab included in the digestion of the calibration standards? YES NO
23. Is a blank analyzed for quality control purposes?
    YES NO
24. Are three elements routinely tested for or is one used as a screen?
    ____ All three
    ____ Screen using one element = Pb Ba Sb
25. What brand of AA do you use? ________________
26. How old is it? ____ year