J Forensic Sci, July 2005, Vol. 50, No. 4
Paper ID JFS2004454
Available online at: www.astm.org

TECHNICAL NOTE

Ludwig Niewoehner,[1] Ph.D.; Jan Andrasko,[2] Ph.D.; Jan Biegstraaten,[3] B.S.; Lawrence Gunaratnam,[4] M.Sc.; Sylvia Steffen,[5] M.Sc.; and Steffen Uhlig,[6] Ph.D.

Maintenance of the ENFSI Proficiency Test Program on Identification of GSR by SEM/EDX (GSR2003)*

ABSTRACT: Within the framework of the ENFSI Expert Working Group "Firearms," every second year, a proficiency test on the detection and identification of GSR by SEM/EDX is carried out. This proficiency test is a development and extension of the previous proficiency test *GSR2001*. The test material was again designed by the organization panel and manufactured by an external company for SEM accessories. This time the participating laboratories were requested to determine the total number of PbSbBa containing particles on a test sample following their own laboratory specific methods of automated GSR particle search and detection by SEM/EDX. One synthetic particle sample (SPS) with artificial GSR particles was dispatched to all participants. This paper summarizes the results of the study and assesses the overall performance of the participating laboratories. Furthermore an extended statistical evaluation and a comparison with previous studies was carried out.

KEYWORDS: forensic science, gunshot residues, GSR, firearms, scanning electron microscopy, SEM/EDX, proficiency testing, European Network of Forensic Science Institutes, ENFSI, ISO 5725, statistical evaluation, z-scores

The detection and identification of gunshot residues (GSR) using scanning electron microscopy and energy dispersive X-ray microanalysis (SEM/EDX) is a well-established technique applied in many forensic science laboratories. This technique is the most reliable in identification of particles consisting of lead, antimony and barium in various proportions, a combination which is commonly accepted as being unique or at least indicative to GSR (1,2,3). In terms of quality assurance, a regular assessment of the automated SEM-search is recommendable.

Within the framework of the Working Group "Firearms" of the European Network of Forensic Science Institutes (ENFSI), a proficiency testing scheme about the detection and identification of GSR by SEM/EDX was set up and performed (4).

Compared with other proficiency tests, where a homogeneous source material can be divided into various split samples for the test, it is a major problem to prepare suitable, i.e., identical test items for a GSR proficiency test. Therefore a technology had to be developed which allowed the preparation of test samples with "artificial" GSR meeting the requirements of proficiency testing according to appropriate standards (4,5,6).

In the past, two proficiency tests have been carried out using synthetically produced GSR particle samples. In the first test (*GSR1999*) samples with synthetic particles consisting of the elements Pb and Sb with three different sizes of 1.2 μm, 2.5 μm and 6.0 μm were distributed to the participants. The second test (*GSR2001*) was an improvement with three-component particles consisting of the elements Pb, Sb and Ba and with particle sizes of 1.0 μm, 2.0 μm and 5.0 μm, respectively (4).

In the present proficiency test (*GSR2003*), again, some improvements were introduced: three-component particles with four different sizes including sub-μm-particles were prepared. In addition to that, a thin carbon layer was applied on the silicon substrate that approaches "real" GSR samples using a carbon-containing adhesive tape. Furthermore, an extensive statistical evaluation of the obtained data was performed by external experts in statistics (7).

Materials and Methods

Preparation of Test Material

The test items for the *GSR2003* proficiency test consisted of specially prepared, identical samples in accordance with the ISO 5725 for the performance of proficiency tests (6). For each of the samples, a total number of 100 "synthetic GSR particles" with the composition PbSbBa were precipitated onto a silicon substrate (size 8×8 mm^2) which was previously applied with a 10 μm carbon layer. The samples were manufactured following a patent protected method for the production of synthetic particles (8). The synthetic

[1] Bundeskriminalamt, BKA, Forensic Science Institute, D-65173 Wiesbaden, Germany.
[2] National Laboratory of Forensic Science, SKL, SE-58194 Linkoeping, Sweden.
[3] Netherlands Forensic Institute, NFI, NL-2497 GB Den Haag, The Netherlands.
[4] National Bureau of Investigation, NBI, PL 285, FIN-01301 Vantaa, Finland.
[5] Tu Freiberg, Institute of Analytical Chemistry, Leipziger Str. 29, D-09599 Freiberg, Germany.
[6] QuoData GmbH, Siedlerweg 20, D-01465 Dresden-Langebrueck, Germany.
* This work was financially supported by the European Union, project no. JAI/2001/OIS/059.
Received 21 Oct. 2004; and in revised form 30 Jan. 2005; accepted 12 March 2005; published 25 May 2005.

Copyright © 2005 by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.

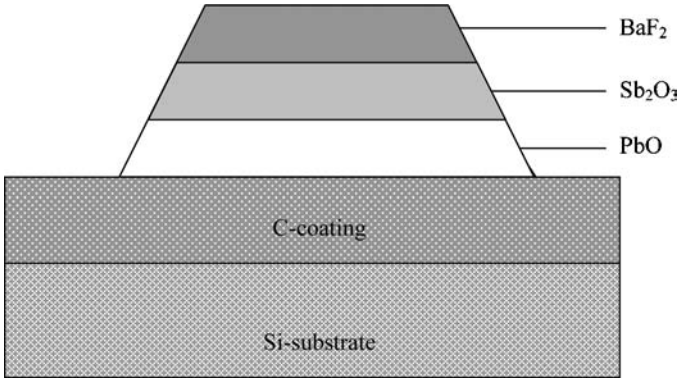

FIG. 1—*Schematic cross-section of a "synthetic GSR-particle."*

TABLE 1—*Size and number of deposited PbSbBa particles on the test samples.*

Sample Description	Total Number of PbSbBa-Particles	2.4 μm Particles	1.2 μm Particles	0.8 μm Particles	0.5 μm Particles
SPS-5P-1A/B/C	100	27	26	25	22

particles resulting from this process show a three-layer structure with the appearance of a frustum. Figure 1 shows a schematic cross-section of such a synthetic GSR-particle. Due to different X-ray excitation volumes regarding the different particle sizes the measured relative elemental compositions of PbSbBa will vary slightly.

The total number of PbSbBa-particles on the surface of the silicon substrate may be higher (so-called etch-resist), but due to the sample production, based on semiconductor process technology (8), the number of "regular" deposited PbSbBa-particles at known locations is fixed to 100. The total number of deposited PbSbBa-particles, their sizes and their location on the sample are well defined. Three different sample layouts were used (samples labeled *SPS-5P-1A*, *SPS-5P-1B* and *SPS-5P-1C*, respectively). Those layouts show only differences in the particle positions, thus still fulfilling the need of identical sample material demanded for proficiency testing. Finally the samples were coated with a thin carbon layer to prevent charging and to protect against mechanical damage.

Table 1 summarizes the relevant information on the test material used for this study.

Homogeneity and Stability of the Test Material

In order to verify the quality of the sample material and to examine possible defects in the number of particles (i.e., missing PbSbBa-particles), a test of homogeneity was carried out. Eight randomly selected samples from each layout were examined manually by SEM/EDX and controlled for completeness. Those 24 samples were taken as a representative basis for the calculation of the lower 95% one-sided confidence limit for the probability that a randomly selected particle does not exist. These lower limits were further used to calculate the probability of *none*, *one* or *two or more* particles of interest missing. On all of these samples no defects were observed. Because all figures are based on confidence limits, the resulting probabilities can be interpreted in terms of lower and upper limits of probabilities of defects. The underlying statistical model is the binomial distribution model. Calculations were performed separately for each size class. Since there were no differences between the three layouts, all the data could be combined into a single set for each particle size class (24 analyzed samples per class). Table 2 shows the resulting probabilities.

TABLE 2—*Lower/Upper limits of probabilities of defects.*

Size Class	No Particle Missing	1 Particle Missing	2 Or More Particles Missing
0.5 μm (22 particles)	>88.265%	<11.049%	<0.686%
0.8 μm (25 particles)	>88.265%	<11.045%	<0.690%
1.2 μm (26 particles)	>88.265%	<11.044%	<0.691%
2.4 μm (27 particles)	>88.265%	<11.043%	<0.692%
all classes (100 particles)	>59.884%	<37.385%	<2.731%

As the probabilities for one missing particle seem to be unacceptably high, it was decided to allow at the most one defect (i.e., missing particle) over all size classes per test sample. More than one defect per size class will only appear with a probability of less than 0.7%. This implies a probability of less than 2.7% that there will be more than one defect in total per sample (at least 99 out of 100 particles present).

The prepared test samples were proven to be long-term stable if properly applied (long-term stable in the sense of the presence of particles, their sizes and elemental proportions).

The Proficiency Test GSR2003

Organization of the Test—The distribution of the test samples to the participating laboratories and the data evaluation were carried out by an external company, QuoData GmbH, under the supervision of the Bundeskriminalamt.

The participating laboratories that submitted their results within deadline are listed in Table 3. Sample classification and data evaluation were performed according to Table 4.

Test samples were sent to 56 laboratories, which had definitely stated their participation in the study and had accepted the conditions for participation. The Bundeskriminalamt received analytical results from 51 laboratories within the pre-set time frame, whereas 8 laboratories analyzed their test sample in various SEM/EDX systems, thus submitting more than one independent result. Three laboratories had to be excluded from evaluation because of insufficient data. Altogether 56 data sets from 48 laboratories were considered in the statistical evaluation of the study.

The participants were requested to carry out a particle search according to their standard parameter settings for automated GSR search by SEM/EDX. In order to avoid edge effects within the automated analysis, particles were only placed in a central area of $7 \times 7\ mm^2$. At least this central area of the test chip had to be searched for particles. The laboratories were not informed about the total number of particles and their sizes. The XY co-ordinates and the sizes of the detected PbSbBa-particles had to be reported, together with the minimum detectable particle size according to the system's parameter settings.

Laboratory Evaluation and Data Assessment—Before starting the statistical evaluation, the submitted raw data had to be cleared of multiply counted particles and of 'non-regular' PbSbBa-particles (etch-resist) by the organization committee as follows: All correctly detected "regular" PbSbBa-particles were displayed in an XY-plot, subtracting the etch-resists while comparing the individual laboratory result data with the master layout. This information was then sent to the participating laboratories in order to give a quick response to the individual laboratory (individual report), and to give the various laboratories the opportunity to cross-read the evaluated results.

The corrected data of all participants are summarized in Table 5. Values in brackets were not considered in the final z-score

TABLE 3—*List of participating laboratories (GSR2003).*

Participating Laboratories (Laboratory Names Sorted Alphabetically)	
Alameda County Sheriff's Crime Lab	U.S.A
Bayerisches LKA, Kriminaltechnisches Institut	Germany
Bundeskriminalamt, KT23	Germany
Carabinieri Investigazioni Scientifiche	Italy
Central Forensic Laboratory of Polish Police	Poland
Ontario Ministry of Public Safety and Security; Centre of Forensic Sciences	Canada
Comisaria General de Policia Cientifica Spain	Spain
Estonian Police Forensic Service Center	Estonia
Forensic Institute	Croatia
Forensic Science Laboratory	Ireland
Forensic Science Northern Ireland	Northern Ireland
Forensic Science Service	United Kingdom
Forensic Science South Australia	Australia
Guardia Civil	Spain
Hamilton County Coroner's Laboratory	U.S.A.
Hessisches Landeskriminalamt—KWTI - FB 711	Germany
Illinois State Police Forensic Science Center at Chicago	U.S.A.
Institute of Criminalistics Prague	Czech Republic
Institute of Forensic Research	Poland
Instituto Nacional de Toxicologia	Spain
IRCGN	France
Israel Police HQ/Div. Of Identification and Forensic Science (DIFS)	Israel
KEU PZ SR, Kriminalisticky a expertizny ustav Pz	Slovakia
Kriminaltechnische Dienste, St. Gallen	Switzerland
KTI/LKA Baden-Württemberg	Germany
Laboratoire de Police Scientifique, Paris	France
Laboratoire de Police Scientifique de Lille	France
Laboratoire de Police Scientifique Lyon	France
Laboratoria de Policia Cientifica—Policia Judiciaria	Portugal
Laboratorio di Scienze Criminalistiche—Universita di Torino	Italy
Laboratory of Forensic Sciences of Marseilles	France
Landeskriminalamt Berlin, Institut Polizeitechnische Untersuchungen LKA PTU 22	Germany
Landeskriminalamt Niedersachsen	Germany
LKA Brandenburg	Germany
LKA Mecklenburg-Vorpommern, Abt. 7, Dez. 73	Germany
LKA Nordrhein-Westfalen	Germany
LKA Rheinland-Pfalz	Germany
LKA Sachsen	Germany
LKA Sachsen-Anhalt	Germany
LKA Thüringen, Dez. 41	Germany
National Bureau of Investigation	Finland
National Criminal Investigation Service	Norway
National Laboratory of Forensic Science (SKL)	Sweden
Netherlands Forensic Institute NFI	The Netherlands
NICC	Belgium
Ohio Bureau of Criminal Identification and Investigation	U.S.A.
Polizei Hamburg, LKA 33	Germany
Reparto Carabinieri Investigazioni Scientifiche Roma	Italy
RIS Carabinieri Parma	Italy
Royal Canadian Mounted Police; Forensic Laboratory Services	Canada
Virginia Division of Forensic Science	U.S.A.

TABLE 4—*Administrative schedule for the GSR2003 study.*

Steps
1. Coding of the participating laboratories, assigned Lab-Codes #001 to #999
2. Validation of the received data
3. Creation of an Excel spread-sheet of all data (raw and corrected)
4. Twofold comparison of the created database with the original data
5. Preparation and distribution of Individual Report for cross-reading of data by the individual laboratory
6. Import of the data into the software package ProLab2003 for statistical evaluation
7. Evaluation of the data according to DIN 38402 A45 (11)

calculations and laboratory assessment because of restrictions in the system's parameter settings for the "minimum particle size detection limit."

The assessment was performed using the software package ProLab2003 (7), which is widely employed for the evaluation of laboratory proficiency tests. The robust statistical Q-method in combination with the Hampel estimator was selected in order to take into account the discrete nature of the data (9,11). This method meets the standard DIN 38402 A45 (11). The statistical theory and background of the Q-method is described in more detail in (12,13). Further statistical aspects concerning the assessment of laboratories are discussed in (14).

TABLE 5—*Corrected data (number of particles) for the 6 measurement characteristics.*

Lab Code #	Size 0.5 µm	Size 0.8 µm	Size 1.2 µm	Size 2.4 µm	Total ≥0.8	Total ≥1.2
# 016	16	23	26	27	76	53
# 028	22	23	26	27	76	53
# 041	(0)	(0)	25	27	(52)	52
# 053	0	0	0	25	25	25
# 078-1	(0)	(0)	8	25	(33)	33
# 078-2	(0)	(0)	7	27	(34)	34
# 089	(0)	(0)	16	26	(42)	42
# 093	(0)	(7)	26	27	(60)	53
# 107	0	3	26	27	56	53
# 119	(0)	(0)	21	27	(48)	48
# 122-1	19	25	25	27	77	52
# 122-2	16	24	25	27	76	52
# 122-3	(11)	21	26	27	74	53
# 134	(0)	(17)	24	27	(68)	51
# 148	0	19	25	27	71	52
# 156	0	0	0	25	25	25
# 165-1	(7)	25	26	27	78	53
# 165-2	(12)	22	26	27	75	53
# 328-1	3	18	26	27	71	53
# 328-2	0	9	23	27	59	50
# 334	3	24	26	27	77	53
# 340-1	17	24	26	27	77	53
# 340-2	20	25	26	27	78	53
# 357	0	0	0	26	26	26
# 368	(7)	(15)	19	23	(57)	42
# 376	0	18	24	25	67	49
# 386	4	18	24	21	63	45
# 395	(0)	(1)	21	26	(48)	47
# 405	(2)	(23)	21	23	(67)	44
# 414	(0)	24	26	27	77	53
# 425	(0)	25	26	27	78	53
# 432	(0)	(0)	0	27	(27)	27
# 441	(0)	21	26	27	74	53
# 456	0	0	11	25	36	36
# 484	22	25	26	27	78	53
# 494	17	23	26	26	75	52
# 508	8	10	11	12	33	23
# 515	(0)	12	26	27	65	53
# 529	(0)	1	13	26	40	39
# 531	(0)	(8)	26	27	(61)	53
# 633	8	23	26	26	75	52
# 642	(0)	(0)	0	26	(26)	26
# 666	0	0	4	24	28	28
# 688	21	22	26	25	73	51
# 697	(0)	(0)	0	6	(6)	6
# 707	21	24	23	25	72	48
# 717	3	11	26	27	64	53
# 722	0	0	16	22	38	38
# 749	0	0	16	26	42	42
# 754	0	9	25	25	59	50
# 769	0	9	12	19	40	31
# 778-1	0	18	26	26	70	52
# 778-2	20	24	26	27	77	53
# 778-3	8	19	26	27	72	53
# 780	0	0	21	25	46	46
# 791	0	18	22	23	63	45

The reference values (X) for the number of PbSbBa-particles for the individual particle sizes were set to one less than the true value, resulting from subtracting an allowed error of one particle as discussed previously. The relative standard deviation was set to an upper limit of 10% of the reference value X. In case of the particle size of 2.4 µm, the empirically determined standard deviation is below this limit. In all other cases the limited standard deviation of 10% was used for further calculations. The values of all characteristics used for the calculation of the z-scores are shown in Table 6.

An assessment of the laboratory's capability to detect GSR particles by SEM/EDX was carried out using z-scores according to IUPAC and EURACHEM (15,16). The z-score of an individual laboratory was calculated by

$$z = \frac{x - X}{S}$$

where x ist the result obtained by the laboratory, X is the "true value," i.e., the correct number of precipitated GSR particles on the sample, and S is the standard deviation.

The z-scores of all evaluated laboratories are given in Table 7. Regarding the characteristic TOTAL ≥1.2 (this value is best comparable to the characteristic TOTAL in the proficiency test *GSR2001* (4)), 35 out of 49 laboratories obtained satisfactory z-scores ($|z| < 2$), whereas the results of 2 laboratories are considered as 'questionable' ($2 \leq |z| \leq 3$). 12 laboratories were considered to have obtained unsatisfactory results ($|z| > 3$). For these 12 laboratories it can be stated with a certainty of at least 95% that the assessment is correct. The obtained values for the six characteristics are given in Table 8.

Table 9 shows the laboratory assessments obtained in the previous proficiency test.

Estimation of the Method's Detection Capability—To estimate the overall quality of the SEM/EDX method in GSR investigation, the method's detection capability was determined. It describes the probability for a randomly selected laboratory to detect a particle of a certain particle size. In order to quantify the detection capability of the method with regard to the particle size, the probability p of detection was modelled according to

$$p = \frac{1}{1 + \left(\frac{A}{m_{50}}\right)^{-s}}$$

In this formula A denotes the particle size whereas the two estimation parameters s and m_{50} describe the steepness (s) and the particle size, for which a detection capability of 50% is interpolated (m_{50}). This model was fitted to all laboratory data using the method of Nonlinear Weighted Least Squares (see e.g., (17)). Weights were calculated based on a binomial distribution.

The calculation was performed iteratively, and starting parameters were obtained using a loglinear regression. This procedure is statistically consistent and asymptotically equivalent to a Maximum Likelihood Analysis. The resulting parameters are $m_{50} = 0.7$ µm and $s = 2.51$, respectively. The corresponding method's detection capability is shown in Figure 2. According to this graph a randomly selected laboratory will detect on average a 1.0 µm particle with a probability of approx. 65%. As a conclusion it can be stated that,

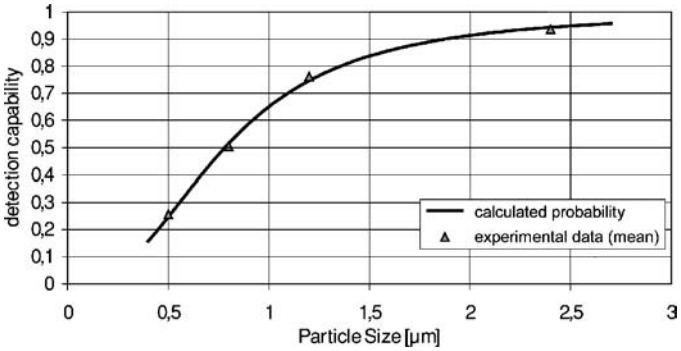

FIG. 2—*Method's detection capability.*

TABLE 6—*Mean values and standard deviations for the different characteristics.*

Measurement Characteristics	Total No. of Particles Per Sample	Reference X^*	Standard Dev. Empirical	Standard Dev. Target (10% of Reference X)	Standard Dev. Used
Total ≥1.2 (No. of det. PbSbBa particles with a diameter of 1.2 μm and 2.4 μm)	53	52	5.4	5.2	5.2
Total ≥0.8 (No. of det. PbSbBa particles with a diameter of 0.8 μm, 1.2 μm and 2.4 μm)	78	77	9.6	7.7	7.7
Size 2.4 (No. of det. PbSbBa particles with a diameter of 2.4 μm)	27	26	1.4	2.6	1.4
Size 1.2 (No. of det. PbSbBa particles with a diameter of 1.2 μm)	26	25	4.4	2.5	2.5
Size 0.8 (No. of det. PbSbBa particles with a diameter of 0.8 μm)	25	24	6.8	2.4	2.4
Size 0.5 (No. of det. PbSbBa particles with a diameter of 0.5 μm)	22	21	6.2	2.1	2.1

* When referring to the value "reference X." It has been calculated from the test of homogeneity and stability, that even for the characteristics Total ≥1.2 and Total ≥0.8 only one missing particle in total is accepted.

on average, 90% of the particles having a minimum size of 1.8 μm will be detected applying this method.

The model allows a satisfying fit of the data and is a close approximation to the mean values of the experimental data set for each of the four particle sizes as shown in Fig. 2. However, all data are based on four particles sizes only, and other models might give a satisfying fit as well. Therefore the two-parametric model applied should be considered as a rough approximation of the detection capability. It should also be stressed that the detection capability represents not necessarily the probability of detection for a single laboratory, but for the whole "population" of laboratories. This is apparent especially for large particle sizes: the majority of laboratories have no problems to detect particles of 1.2 μm or 2.4 μm. Many of them have an individual detection capability higher than 95%.

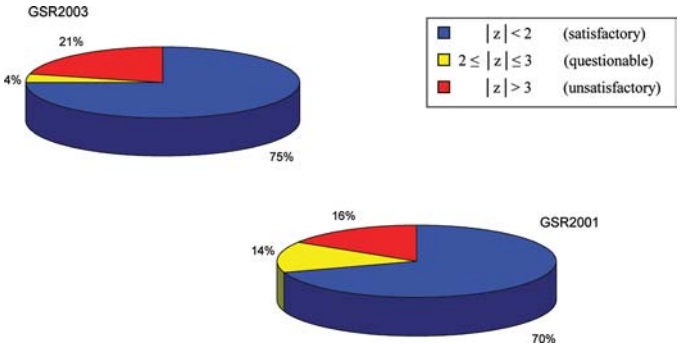

FIG. 3—*Laboratory assessments of GSR2001 and GSR2003.*

Discussion

First of all, this proficiency-testing program is not supposed to be a competition between laboratories, but a promotion of quality in the detection and identification of GSR by automated SEM/EDX analysis. As the participating laboratories were additionally requested to submit also their SEM/EDX operating conditions, the organizing committee was able to give some advise concerning modified parameter settings for an improved system performance (4).

In the current study, sub-μm particles have been introduced as a new challenge for the participating laboratories and their SEM/EDX systems. It could be shown that some laboratories are capable to detect even particles as small as 0.5 μm with their standard instrument settings routinely. However there are other laboratories using instrumental settings unsuitable for the detection of sub-μm particles. This has been considered in the evaluation of the laboratory assessments for the different characteristics, but not in the previous tests. A direct comparison of the *GSR2003* test with the previous test (*GSR2001*) (4) is therefore difficult. But even though there have been made some slight modifications in the sample design, the obtained laboratory assessments show similar results (see Tables 8 and 9). Taking into account the characteristic TOTAL from the *GSR2001* and the TOTAL ≥1.2 from the *GSR2003*, the success rates are 70% and 75%, respectively (see Figure 3). The further characteristics are not suitable for comparison because of significant differences in the particle size.

The new sample design consisting of 4 different particle size classes facilitates an estimation of the overall method's detection capability as shown in Fig. 2. It shows the detection capability as a function of the particle size. This, for the first time, allows a validation of the performance of the SEM/EDX system as a standard method for the investigation of GSR.

As expected, the method's detection capability decreases with an decrease in particle size. To achieve a detection capability of at least 90%, the particle size has to exceed 1.8 μm. For 1.0 μm particles the detection capability is only 65% at present. Using the existing equipment, it seems to be possible to improve the performance of the method by changing the standard parameter settings in certain laboratories.

The aim of future proficiency tests will be a further optimization of the parameter settings in order to enhance the sensitivity of the method and to harmonize the performance of the GSR investigation in various laboratories.

TABLE 7—*Calculated z-scores for the 6 measurement characteristics.*

Lab Code #	Size 0.5 µm	Size 0.8 µm	Size 1.2 µm	Size 2.4 µm	Total ≥0.8	Total ≥1.2
# 016	−2.4	−0.4	0.4	0.7	−0.1	0.2
# 028	0.5	−0.4	0.4	0.7	−0.1	0.2
# 041			0.0	0.7		0.0
# 053	−10.0	−10.0	−10.0	−0.7	−6.8	−5.2
# 078-1			−6.8	−0.7		−3.7
# 078-2			−7.2	0.7		−3.5
# 089			−3.6	0.0		−1.9
# 093			0.4	0.7		0.2
# 107	−10.0	−8.8	0.4	0.7	−2.7	0.2
# 119			−1.6	0.7		−0.8
# 122-1	−1.0	0.4	0.0	0.7	0.0	0.0
# 122-2	−2.4	0.0	0.0	0.7	−0.1	0.0
# 122-3		−1.3	0.4	0.7	−0.4	0.2
# 134			−0.4	0.7		−0.2
# 148	−10.0	−2.1	0.0	0.7	−0.8	0.0
# 156	−10.0	−10.0	−10.0	−0.7	−6.8	−5.2
# 165-1		0.4	0.4	0.7	0.1	0.2
# 165-2		−0.8	0.4	0.7	−0.3	0.2
# 328-1	−8.6	−2.5	0.4	0.7	−0.8	0.2
# 328-2	−10.0	−6.3	−0.8	0.7	−2.3	−0.4
# 334	−8.6	0.0	0.4	0.7	0.0	0.2
# 340-1	−1.9	0.0	0.4	0.7	0.0	0.2
# 340-2	−0.5	0.4	0.4	0.7	0.1	0.2
# 357	−10.0	−10.0	−10.0	0.0	−6.6	−5.0
# 368			−2.4	−2.2		−1.9
# 376	−10.0	−2.5	−0.4	−0.7	−1.3	−0.6
# 386	−8.1	−2.5	−0.4	−3.6	−1.8	−1.3
# 395			−1.6	0.0		−1.0
# 405			−1.6	−2.2		−1.5
# 414	−5.7	0.0	0.4	0.7	0.0	0.2
# 425		0.4	0.4	0.7	0.1	0.2
# 432			−10.0	0.7		−4.8
# 441		−1.3	0.4	0.7	−0.4	0.2
# 456	−10.0	−10.0	−5.6	−0.7	−5.3	−3.1
# 484	0.5	0.4	0.4	0.7	0.1	0.2
# 494	−1.9	−0.4	0.4	0.0	−0.3	0.0
# 508	−6.2	−5.8	−5.6	−10.2	−5.7	−5.6
# 515		−5.0	0.4	0.7	−1.6	0.2
# 529		−9.6	−4.8	0.0	−4.8	−2.5
# 531			0.4	0.7		0.2
# 633	−6.2	−0.4	0.4	0.0	−0.3	0.0
# 642			−10.0	0.0		−5.0
# 666	−10.0	−10.0	−8.4	−1.5	−6.4	−4.6
# 688	0.0	−0.8	0.4	−0.7	−0.5	−0.2
# 697			−10.0	−14.6		−8.8
# 707	0.0	0.0	−0.8	−0.7	−0.6	−0.8
# 717	−8.6	−5.4	0.4	0.7	−1.7	0.2
# 722	−10.0	−10.0	−3.6	−2.9	−5.1	−2.7
# 749	−10.0	−10.0	−3.6	0.0	−4.5	−1.9
# 754	−10.0	−6.3	0.0	−0.7	−2.3	−0.4
# 769	−10.0	−6.3	−5.2	−5.1	−4.8	−4.0
# 778-1	−10.0	−2.5	0.4	0.0	−0.9	0.0
# 778-2	−0.5	0.0	0.4	0.7	0.0	0.2
# 778-3	−6.2	−2.1	0.4	0.7	−0.6	0.2
# 780	−10.0	−10.0	−1.6	−0.7	−4.0	−1.2
# 791	−10.0	−2.5	−1.2	−2.2	−1.8	−1.3

TABLE 8—*Overall proficiency values for the 6 characteristics (GSR2003).*

	Satisfactory ($\|z\| < 2$)	Questionable ($2 \le \|z\| \le 3$)	Unsatisfactory ($\|z\| > 3$)
TOTAL ≥1.2	42 (75%)	2 (4%)	12 (21%)
TOTAL ≥0.8	28 (67%)	3 (7%)	11 (26%)
SIZE 2.4 µm	48 (86%)	4 (7%)	4 (7%)
SIZE 1.2 µm	39 (70%)	1 (2%)	16 (28%)
SIZE 0.8 µm	19 (45%)	7 (17%)	16 (38%)
SIZE 0.5 µm	9 (26%)	2 (6%)	24 (68%)

TABLE 9—*Overall proficiency values for the 3 characteristics (GSR2001).*

	Satisfactory ($\|z\| < 2$)	Questionable ($2 \le \|z\| \le 3$)	Unsatisfactory ($\|z\| > 3$)
TOTAL	31 (70%)	6 (14%)	7 (16%)
SIZE 2.0 µm	29 (66%)	5 (11%)	10 (23%)
SIZE 1.0 µm	31 (70%)	3 (7%)	10 (23%)

Acknowledgment

The authors would like to thank the European Union for financial support of the study (EU-Project No. JAI/2001/OIS/059).

References

1. Meng HH, Caddy B. Gunshot residue analysis—review. J Forensic Sci 1997;42:553–70.
2. Romolo FS, Margot P. Identification of gunshot residue: a critical review. Forensic Sci Int 2001;119:195–211. [PubMed]
3. ASTM Guide E-1588-95 (reapproved in 2001), Standard guide for gunshot residue analysis by scanning electron microscopy/energy-dispersive spectroscopy. West Conshohocken: ASTM International 2002;14.02.
4. Niewoehner L, Wenz W, Andrasko J, Beijer R, Gunaratnam L. ENFSI proficiency test program on identification of GSR by SEM/EDX. J Forensic Sci 2003;48(4):786–92. [PubMed]
5. ISO Guide 43-1 & -2:1996(E): Proficiency testing by interlaboratory comparisons—part 1: development and operation of proficiency testing schemes—part 2: selection and use of proficiency testing schemes by laboratory accreditation bodies. Geneva: International Standards Organisation, 2003.
6. ISO 5752-2:1994(E): Accuracy (trueness and precision) of measurement methods and results—part 2: basic method for the determination of repeatability and reproducibility of a standard measurement method. Geneva: International Standards Organisation, 2003.
7. PROLAB 2003: QuoData GmbH; Dresden/Germany, www.quodata.de
8. Niewoehner L, Wenz HW, inventors. Niewoehner L, assignee. Verfahren zur Herstellung synthetischer Partikelproben. Patent DE 199 32 357. 2001 Feb 8.
9. Huber PJ. Robust statistics. New York: John Wiley & Sons, 1981.
10. Rousseeuw PJ. Tutorial to robust statistics. J Chemometrics 1991;5:1–20.
11. DIN 38402 A45-2003, Part 45: Interlaboratory comparisons for proficiency testing of laboratories. Berlin: Beuth Verlag, 2003.
12. Uhlig S. Optimum two-way nested designs for estimation of variance components. Tatra Mt Math Publ 1996;7:105–22.
13. Mueller C, Uhlig S. Estimation of variance components with high breakdown point and high efficiency. Biometrika 2001;88(2):353–66.
14. Uhlig S, Lischer P. Statistically based performance characteristics in laboratory performance studies. The Analyst 1998;123:167–72.
15. Thomson M, Wood R. The international harmonized protocol for the proficiency testing of (chemical) analytical laboratories (technical report). Pure Appl Chem 1993;65:2123–44.
16. EURACHEM guide on selection, use and interpretation of proficiency testing (pt) schemes by laboratories, edition 1.0–2000, www.eurachem.bam.de .
17. McCullagh P, Nelder JA. Generalized linear models. London: Chapman and Hall, 1989.

Additional information and reprint requests:
Ludwig Niewoehner, Ph.D.
Bundeskriminalamt
Forensic Science Institute, KT23
Wiesbaden, 65173
Germany
Phone: +49-611-551 2678
Fax: +49-611-554 5087
E-mail: Ludwig.Niewoehner@bka.bund.de