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ENFSI Proficiency Test Program on Identification of GSR by SEM/EDX*

ABSTRACT: Within the framework of the ENFSI Expert Working Group "Firearms," a proficiency test on the detection and identification of GSR by SEM/EDX was organized and performed. The test material was designed by the Bundeskriminalamt and manufactured to order by an external company for SEM accessories. The participating laboratories were requested to determine the total number of PbSbBa-containing particles on the test samples following their own laboratory specific methods of automated GSR particle search and detection by SEM/EDX. Two similar samples with synthetic GSR particles were dispatched to all participants in order to gain additional information on systematic errors within the obtained results (split-level study), whereas one sample was supplied only with PbSbBa particles, and the second one was additionally contaminated with some environmental particles. This report summarizes the results of the study as well as a statistical evaluation and comparison with previous studies.

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, Youden, 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, a combination that is commonly accepted as being unique to GSR (1–3). Manually searching for GSR has almost completely been replaced by automated GSR search systems that require a frequently performed careful validation of the analytical procedure.

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. In 1995, at the second annual ENFSI meeting of the Working Group, it was decided to start a proficiency-testing program for members of the working group. The aim of this program was not a competition between laboratories, but the promotion of quality in the detection and identification of GSR by automated SEM/EDX analysis.

Proficiency testing programs have been introduced to different areas of forensic science investigations such as fibers, glass, paint flakes, and DNA for a long time, but only some of them have been published (4–6). In the past, the company CTS Inc. performed several collaborative studies and GSR research tests on the detection and identification of GSR. There is, however, a significant problem in the preparation of GSR test samples that meet the necessary requirements of a proficiency test. Compared to other proficiency tests, where, e.g., 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. In the preparation of test samples with "real" GSR, there will always be a statistical variance in the total number of particles deposited on the surface of a stub as well as a variation in the diameter of the GSR particles and their chemical composition. It is, of course, possible to use one GSR sample (SEM stub provided with an adhesive tape) that is initially carefully checked by the organizer of the study and then distributed in succession to all participating laboratories for investigation (round-robin study). Such a test would be very time consuming because of the necessity of a permanent sample quality control. Furthermore, there is also a risk of possible sample modifications in the number of particles and contamination or loss of particles during the performance of the study.

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Materials and Methods

Preliminary Studies

In the first collaborative study, performed in 1996, GSR samples were prepared using real GSR particles produced by a single shot on a sheet of medical gauze, removed in an ultrasonic bath using ethanol, and diluted to a useable concentration of GSR particles. An aliquot of 20 mL of this solution was pressed through a two-stage polycarbonate microfilter system with pore sizes of 10 and 0.8 μm, respectively. The smaller pore size filter was then mounted on a half-inch aluminum stub and coated with a thin carbon layer. All prepared samples were first examined in the Forensic Science Institute of the Bundeskriminalamt before they were distributed to the participating laboratories (15 in total). The significance of this first test, which was presented at the 3rd ENFSI meeting in Paris (7), was strongly limited. Particularly the fact that the distributed sample material showed a big variance in the total number of GSR particles made an assessment of the results more difficult (Fig. 1). These difficulties were also enhanced by significant differences in the average elemental composition of the GSR particles as well as in their diameters ranging from about 10 down to 0.8 μm.

Due to these problems with real GSR particles, it was decided to modify the sample material for the proficiency test. Instead of samples containing "real" GSR particles, a series of samples with synthetic particles with a composition similar to those of GSR had to be prepared. A technology was used that allows the production of an unlimited number of test samples with a known number of GSR particles with well-defined diameters, known elemental compositions, and known locations on the sample surface. A more detailed description of the production process is described later in this study.

In a second study, called "ENFSI Proficiency Test on Identification of GSR by SEM/EDX," samples containing synthetic GSR particles of three different sizes (1.2, 2.5, and 6 μm in diameter) were prepared. Each of the participating laboratories received one sample with synthetic particles containing lead and antimony. The number of particles, their composition, size, and position were known and were exactly the same for each of these samples. The size and number of particles are shown in Table 1.

The study was carried out in the years 1999/2000 (subsequently cited as "GSR1999") and produced overall good results. A total of 46 laboratories participated in this proficiency test, whereas three laboratories submitted two independent results from different SEM/EDX systems. As a result, 49 datasets were considered in the final assessment. The assessment of the laboratories was obtained using z-scores (8,9). The z-score of an individual laboratory was calculated by:

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

where x is 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 calculated from all received data. Assuming that the results follow a normal distribution, z represents a standardized variable, where a z-score of $|z| > 2$ is expected in only 4.55% of the cases and $|z| > 3$ in only 0.27%. For the assessment of a laboratory a z-score of $|z| > 3$ indicates an unacceptable poor performance of the test, while for satisfactory performance a z-score of $|z| < 2$ is required. Table 2 shows the obtained z-score data of this study.

The evaluation of the GSR1999 study showed that the method of using synthetic particles instead of real ones was promising. The main drawback of this first study with synthetic GSR particles was that the particles consisted only of two elements (lead and anti-

TABLE 1—*Size and number of deposited PbSb particles on the test samples (GSR1999).*

Particle Diameter	No. of PbSb Particles
6 μm	3
2.5 μm	20
1.2 μm	20

TABLE 2—*Summarizing results of the z-score evaluation of the laboratories participating in the GSR1999 proficiency test. The table shows the overall success rate of the participating laboratories.*

Characteristic	Satisfactory ($|z| < 2$)	Questionable ($2 \leq |z| \leq 3$)	Unsatisfactory ($|z| > 3$)
Total*	16 (33%)	7 (15%)	25 (52%)
2 μm†	22 (46%)	14 (29%)	12 (25%)
1 μm‡	13 (27%)	4 (8%)	31 (65%)

* Mean: 43 particles; std. dev.: 2.5 particles.
† Mean: 20 particles; std. dev.: 1 particle.
‡ Mean: 20 particles; std. dev.: 1 particle.

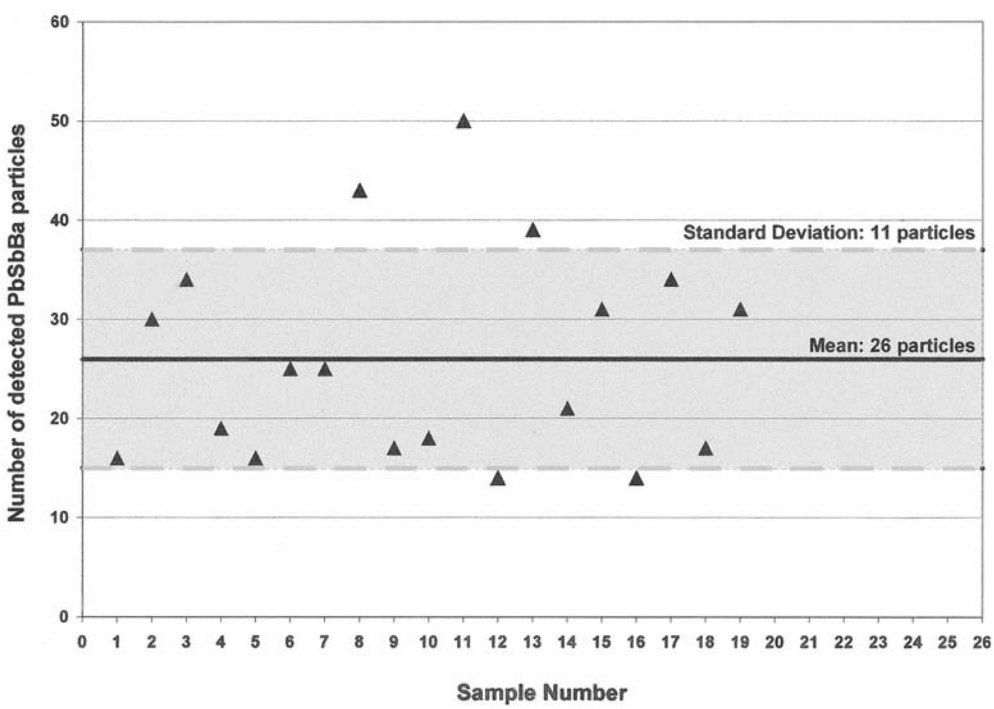

FIG. 1—*Precheck of sample material; number of detected PbSbBa particles on the prepared SEM stubs.*

TABLE 3—*Number of regular particles deposited on the different samples used in the study GSR2001.*

Sample Description	Total Number of PbSbBa Particles	Number of PbSbBa particles with a diameter of:		
		5 μm	2 μm	1 μm
SPS-A (clean)	43	3	22	18
SPS-B (contaminated)	43	3	15	25

mony) because of problems incorporating barium into the particles. The results of this study were reported and published at several international meetings including the 6th Meeting of the ENFSI Expert Working Group "Firearms" and SCANNING (10,11).

The Proficiency Test "GSR2001"

Selection and Preparation of Suitable Sample Material—The test items for the GSR2001 proficiency test consisted of a set of identical samples in accordance with the ISO Guide 43-2 and ISO 5725-2 for the performance of proficiency tests (8,12). The production of the test material was carried out applying selected semiconductor technology processes (13). For each of these samples, "synthetic GSR particles" consisting of PbSbBa were precipitated onto a silicon substrate (8 by 8 mm^2). The total number of PbSbBa particles on the surface of the silicon substrate may be higher due to etch-resist particles, but due to the production process the number of "regular" PbSbBa particles is supposed to be fixed. Then, the substrates were mounted on a standard half-inch stub as commonly used in GSR investigation. The total number of deposited "regular" PbSbBa particles, their size, and their location on the sample were well defined. Finally, the samples were coated with a thin photo-resist layer to avoid mechanical damage (both sample types, i.e., SPS-A and SPS-B). Half of the samples were additionally provided with environmental particles of pure Pb, Fe, and Cu (contaminated sample, i.e., SPS-B). Table 3 summarizes the information on the test materials used for the study.

A test of uniformity was carried out at the Forensic Science Institute of the Bundeskriminalamt, checking the number of PbSbBa particles on a random selection of 60% of the test samples. Most of the controlled samples were proved to have a total number of 43 PbSbBa particles, whereas 11% of the controlled samples showed a deficit of one particle. It was decided that the test material was sufficiently homogeneous for the intended use (43 particles [-1 particle $\cong$ 2% at most]).

Organization of the Test—The distribution of the test samples to the participating laboratories and the data evaluation were carried out by the Bundeskriminalamt. The participating laboratories are listed in Table 4. Table 5 shows the time schedule of the study. Sample sets of two samples (Types SPS-A and SPS-B) were sent to 48 laboratories together with data report sheets and a method description questionnaire. The Bundeskriminalamt received analytical results from 43 laboratories before deadline, whereas three laboratories submitted two independent results from different SEM/EDX systems. Four laboratories had to be excluded because of delayed data submission, and two laboratories submitted insufficient data. Altogether 44 datasets from 41 laboratories were considered in the statistical evaluation of the test.

The participants were requested to carry out an automated GSR particle search by SEM/EDX using their standard parameter settings for each of the two samples. At least 7 by 7 mm^2 of the sample area had to be searched for particles, and the XY co-ordinates as well as the sizes of the detected PbSbBa particles had to be reported.

Before starting the statistical evaluation, multiple counted particles and PbSbBa particles that were detected on nonregular locations ("etch-resist") had to be rejected from the submitted raw data. The data assessment followed the ISO 5725-2 protocol implemented in the applied software PROLAB 2000 (14). Table 6 shows the different steps of the assessment.

The corrected data of all participants are summarized in Table 7. The participating laboratories are represented by their anonymous lab IDs. Figure 2 shows the histogram plot for the evaluated level: total number of detected PbSbBa particles. In the evaluation scheme, three main characteristics were established to look for the two different sample types (clean sample and contaminated sample), which were: *total number of correctly detected PbSbBa particles*, *number of correctly detected 2-μm PbSbBa particles*, and *number of correctly detected 1-μm PbSbBa particles.* A test for outliers was neglected because robust statistics were applied for the statistical assessment of the received data.

The mean value (X_{total}) for the total number of PbSbBa particles was calculated from the test of uniformity to 42.9 particles and rounded off to the "true value" of 43. The standard deviation (S_{total}) was empirically determined based on the achieved valid data. The values for mean and standard deviation used for the calculation of the z-scores are presented in Table 8.

Laboratory Evaluation and Data Assessment—An evaluation of the laboratory's proficiency to detect GSR particles by SEM/EDX was carried out using z-scores according to IUPAC and EURACHEM (9,15,16). Z-scores were calculated using standard deviation and mean as shown in Table 8. The calculated z-scores of all laboratories are given in Table 7. Tables 9*a* to 9*c* summarize the resulting z-score distributions for the samples SPS-A and SPS-B separately as well as together for the final assessment of a laboratory's proficiency.

In Table 9*b*, the Sample SPS-B (contaminated) has been chosen to allow a comparison between the current proficiency test results and the previous one, GSR1999. Regarding the level "total number of detected PbSbBa particles," 75% of the laboratories (33 out of 44 laboratories) obtained satisfactory z-scores ($|z| < 2$), whereas the results of 11% of the laboratories were considered as "questionable" ($2 \leq |z| \leq 3$). Fourteen percent of the participating laboratories were considered to have obtained unsatisfactory results ($|z| > 3$). In the GSR1999 study (see Table 2), only 33% of the laboratories were considered as "satisfactory," whereas 52% showed "unsatisfactory" results.

Additionally, the intralaboratory reproducibility of the participating laboratories was determined by the graphical presentation of the results of both samples (SPS-A and SPS-B) in a Youden plot

TABLE 4—*List of participating laboratories (GSR2001).*

	Agency Names	Country
1	Alameda G. Sheriff's Office Crime Lab	U.S.A
2	Bayerisches Landeskriminalamt	Germany
3	Bundeskriminalamt	Germany
4	Bundesministerium für Inneres der Republik Österreich; Kriminaltechnische Zentralstelle	Austria
5	Central Forensic Laboratory of the Polish Police; Chemistry Department	Poland
6	Centre of Forensic Sciences	Canada
7	Centro de Investigación y Criminalística; Dirección General de la Guardia Civil	Spain
8	Comisaría General de Policía Científica; Laboratorio Químico	Spain
9	Contra Costa Crime Lab	U.S.A
10	Danish Technical Institute	Denmark
11	ESF Environmental Chemistry, Science & Forestry	U.S.A
12	Forensic Institute Bratislava; Dept. of Chemistry	Slovakia
13	Forensic Science Laboratory, Garda HQ	Ireland
14	Forensic Science Northern Ireland	Northern Ireland
15	Forensic Science Service; Birmingham Laboratory	England
16	Hamilton County Coroner's Lab; Chief of Forensic Sciences	U.S.A
17	Hessisches Landeskriminalamt	Germany
18	Honolulu Police Dept.; Scientific Investigation Section	U.S.A
19	I.R.C.G.N.; Département Microanalyse	France
20	Illinois State Police; Forensic Science Center at Chicago	U.S.A
21	Institute of Criminalistics Prague	Czech Republic
22	Institute of Forensic Research; Department of Criminalistics	Poland
23	Toolmarks and Materials Laboratory; Div. of Identification and Forensic Science (DIFS)	Israel
24	Laboratorio de Policia Cientifica; Policia Judiciaria	Portugal
25	Landeskriminalamt Baden-Württemberg	Germany
26	Landeskriminalamt Brandenburg	Germany
27	Landeskriminalamt Hamburg	Germany
28	Landeskriminalamt Mecklenburg-Vorpommern	Germany
29	Landeskriminalamt Niedersachsen	Germany
30	Landeskriminalamt Nordrhein-Westfalen	Germany
31	Landeskriminalamt Sachsen	Germany
32	Landeskriminalamt Sachsen-Anhalt	Germany
33	Landeskriminalamt Schleswig-Holstein	Germany
34	Landeskriminalamt Thüringen	Germany
35	Ministerio de Justicia; Instituto Nacional de Toxicología	Spain
36	Ministry of Interior; Forensic Institute Laboratory for SEM	Croatia
37	Ministry of the Interior	Republic of Slovenia
38	National Bureau of Investigation (NBI)	Finland
39	National Criminal Investigation Service; Laboratory Division	Norway
40	National Laboratory of Forensic Science (SKL); Department of Chemistry and Technology	Sweden
41	Netherlands Forensic Science Institute	The Netherlands
42	NICC; Chemical Ballistics Unit	Belgium
43	Orange County Sheriff's Department; Forensic Science Services	U.S.A
44	Orange County Sheriff's Department; Forensic Science Services	U.S.A
45	Physikalisch-Technische Untersuchungsstelle Berlin	Germany
46	Reparto Carabinieri Investigazioni Scientifiche	Italy
47	Royal Canadian Mounted Police; Forensic Laboratory Services	Canada
48	West Virginia State Police; Forensic Laboratory	U.S.A

TABLE 5—*Time schedule of the study for the determination of GSR by SEM/EDX (GSR2001).*

Date	Action
May 2001	Announcement of the intercomparison study by the ENFSI Working Group "Firearms" Call for statement of participation
Aug. 2001	Distribution of the samples to the participants
Sept. 2001	Deadline for submission of analytical results
Nov. 2001	Dispatch of individual results
Dec. 2001	Statistical evaluation of results; preparation of final report
Jan. 2002	Dispatch of final report

TABLE 6—*Data assessment for the evaluation of the GSR determination study (GSR2001).*

Step	Laboratory
Coding of participating laboratories	Lab-ID #001 to #062
Coding of the available sample material (SPS-A and SPS-B)	Sample-ID #001 to #120
Correction of the received data	…
Creation of an Excel® spread-sheet of all data (raw and corrected)	…
Twofold comparison of the created database with the original data	…
Import of the data into the software package for statistical evaluation	…
	…
Evaluation of the data according to ISO 5725-2	…

TABLE 7—*Corrected data and z-scores for the level "total number of detected PbSbBa particles" (GSR2001).*

	Total Number of Detected Particles		Calculated z-Scores	
Lab Code	Sample SPS-A	Sample SPS-B	Sample SPS-A	Sample SPS-B
#001	43	40	0.0	−1.0
#002 (SEM1)	42	43	−0.4	0.0
#002 (SEM2)	43	43	0.0	0.0
#003	41	43	−0.9	0.0
#004	43	43	0.0	0.0
#006	43	43	0.0	0.0
#007	41	41	−0.9	−0.7
#008	40	43	−1.3	0.0
#009 (SEM1)	43	43	0.0	0.0
#009 (SEM2)	43	43	0.0	0.0
#010	39	39	−1.7	−1.3
#011	37	32	−2.6	−3.7
#012 (SEM1)	43	43	0.0	0.0
#012 (SEM2)	42	42	−0.4	−0.3
#013	42	41	−0.4	−0.7
#014	43	43	0.0	0.0
#020	0	1	−18.7	−14.2
#021	43	43	0.0	0.0
#022	32	26	−4.8	−5.7
#023	32	34	−4.8	−3.0
#025	43	31	0.0	−4.0
#026	36	35	−3.0	−2.7
#027	43	43	0.0	0.0
#028	38	39	−2.2	−1.3
#029	22	20	−9.1	−7.8
#031	38	41	−2.2	−0.7
#034	43	43	0.0	0.0
#035	32	35	−4.8	−2.7
#036	41	39	−0.9	−1.3
#038	31	39	−5.2	−1.3
#039	42	42	−0.4	−0.3
#040	42	42	−0.4	−0.3
#041	43	43	0.0	0.0
#050	22	30	−9.1	−4.4
#051	36	38	−3.0	−1.7
#052	43	36	0.0	−2.4
#053	42	43	−0.4	0.0
#054	37	38	−2.6	−1.7
#055	41	40	−0.9	−1.0
#057	39	36	−1.7	−2.4
#058	43	42	0.0	−0.3
#059	41	40	−0.9	−1.0
#060	43	43	0.0	0.0
#062	40	38	−1.3	−1.7

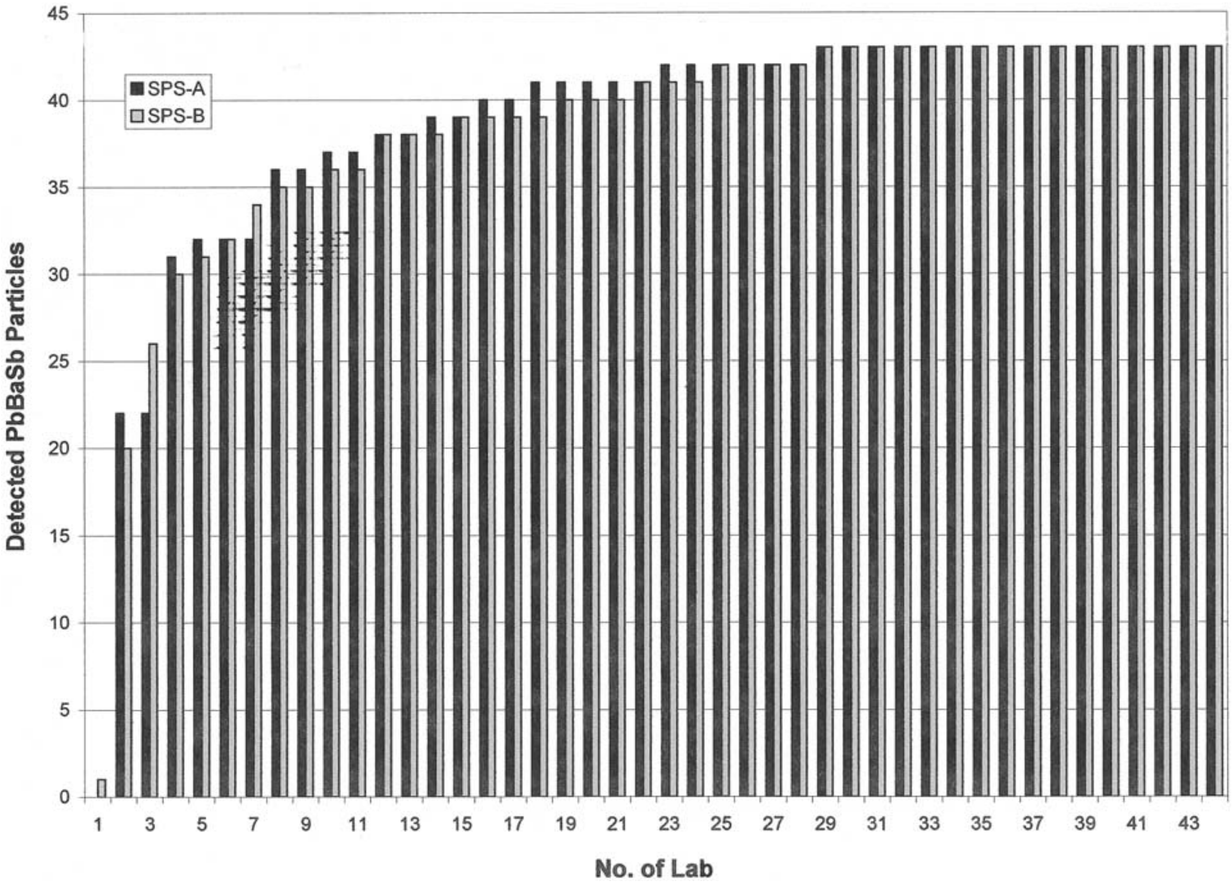

FIG. 2—*Histogram plot for the level "total number of detected PbSbBa particles."*

TABLE 8—*Mean values and standard deviations for the different levels and samples (GSR2001).*

	SPS-A		SPS-B	
Level	Mean	Std. Dev.	Mean	Std. Dev.
Total number of detected PbSbBa particles	43	2.302	43	2.966
Number of detected PbSbBa particles with a diameter of 2 μm	22	0.869	15	0.622
Number of detected PbSbBa particles with a diameter of 1 μm	18	1.173	25	1.824

(17,18) (Fig. 3). This plot reveals precision and accuracy of a certain laboratory and allows an estimation of systematic errors. A Youden plot illustrates intra- and interlaboratory success rates at the same time.

Discussion

When a preliminary test on the detection and identification of GSR by SEM/EDX was carried out, problems with finding the "real" number of GSR particles have been encountered. The lack of correspondence in the obtained results could not be explained by objective reasons such as statistical variation. This led to the con-

TABLES 9a to 9c—*Summarizing results of the z-score evaluation of the laboratories participating in the GSR2001 proficiency test. The tables show the overall success rate of the participating laboratories regarding the separate samples (a, b) and both samples together (c).*

TABLE 9*a*

Characteristic (Type SPS-A)	Satisfactory ($\|z\| < 2$)	Questionable ($2 \leq \|z\| \leq 3$)	Unsatisfactory ($\|z\| > 3$)
Total	31 (70%)	6 (14%)	7 (16%)
2 μm	29 (66%)	5 (11%)	10 (23%)
1 μm	31 (70%)	3 (7%)	10 (23%)

TABLE 9*b*

Characteristic (Type SPS-B)	Satisfactory ($\|z\| < 2$)	Questionable ($2 \leq \|z\| \leq 3$)	Unsatisfactory ($\|z\| > 3$)
Total	33 (75%)	5 (11%)	6 (14%)
2 μm	29 (66%)	0 (0%)	15 (34%)
1 μm	33 (75%)	4 (9%)	7 (16%)

TABLE 9*c*

Characteristic (Types SPS-A, SPS-B)	Satisfactory ($\|z\| < 2$)	Questionable ($2 \leq \|z\| \leq 3$)	Unsatisfactory ($\|z\| > 3$)
Total	28 (64%)	7 (16%)	9 (20%)
2 μm	24 (55%)	3 (7%)	17 (38%)
1 μm	27 (61%)	6 (14%)	11 (25%)

clusion that some automated particle search systems may not operate properly and have to be validated. Therefore, an approach has been made designing an appropriate sample specimen as demanded in the ISO 5725 for the performance of proficiency tests meeting the requirements of identical/homogeneous test items. Those requirements are: known number, composition, location, and size of the particles of interest.

This evaluation was focused on the achievements of the automatic particle search system concerning different aspects. Three main characteristics were established to look for the two different sample types (clean sample and contaminated sample), which were: *total number of correctly detected PbSbBa particles, number of correctly detected 2-μm PbSbBa particles, and number of correctly detected 1-μm PbSbBa particles.* Following these aspects, a characterization of systematic and random errors could be verified additionally by means of a Youden plot and by calculating z-scores.

The evaluation according to Youden permits not only information on the degree of systematic errors within a laboratory (intralaboratory precision), but also allows a determination of precision and accuracy between different laboratories and their automatic systems.

Before starting the statistical evaluation, a critical look on the submitted data had to be taken. Participants may have reported a smaller number of detected GSR particles than those really present on the sample. The number of reported GSR particles could also be too large.

The loss and/or surplus of GSR particles present may be attributed to several reasons:

- Inadequate focusing of the electron beam, which causes some particles (particularly the smaller ones) to be lost for detection.
- Improper threshold value applied to the backscattered electron signals that may result in a loss of particles.
- Inadequate adjusted frame by frame movements of the stage. Multiple detected particles can be found when the areas scanned in two adjacent frames are overlapping. A loss of particles will appear when there is spacing between these two frames. This phenomenon may appear in the x- or/and in the y-direction. In addition to these factors, the different scanning rates may also influence the number of detected particles.

Considering these phenomena, finding correlations between the obtained results and the parameter settings within the used SEM/EDX systems was attempted. From these examinations, basic information on system dependent parameter settings for an adequate analysis of GSR could be obtained.

When comparing the latest results with those from the previous proficiency test, it is obvious that there has been an improvement concerning the detection of the total number of detected particles. More laboratories use optimized parameter settings with their systems and therefore detect in most cases at least 90% of the total number of PbSbBa particles. Moreover, it can already be claimed that the error of losing mainly the smaller particles (<2 μm) has decreased and is now statistically distributed. This effect is confirmed in the diagram "Cluster Frequencies of the Lab Results" (Fig. 4).

In general, the aim of a proficiency test is not to contest an event but to offer a way to reveal eventual insufficiencies in particle detection arising in the different analytical systems. With the assistance of trained personnel from the organization panel of the proficiency test, it should be possible to minimize or even remove systematic errors in the future. It might also be of interest for the

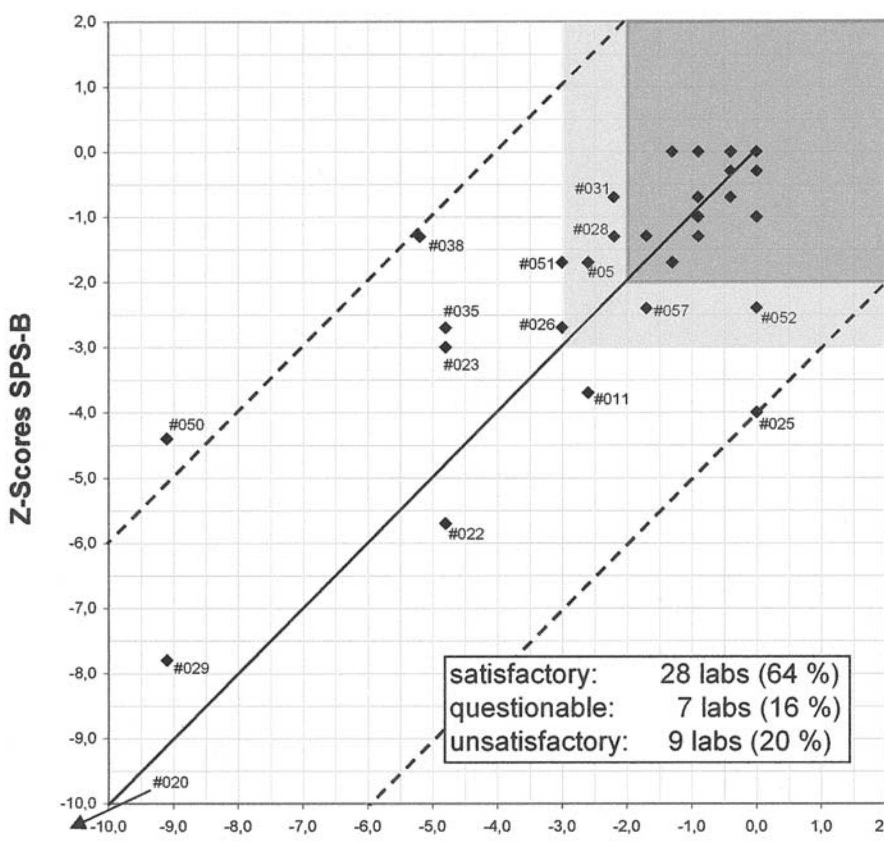

FIG. 3—*Youden plot for the characteristic "total number of detected PbSbBa particles" (SPS-A and SPS-B).*

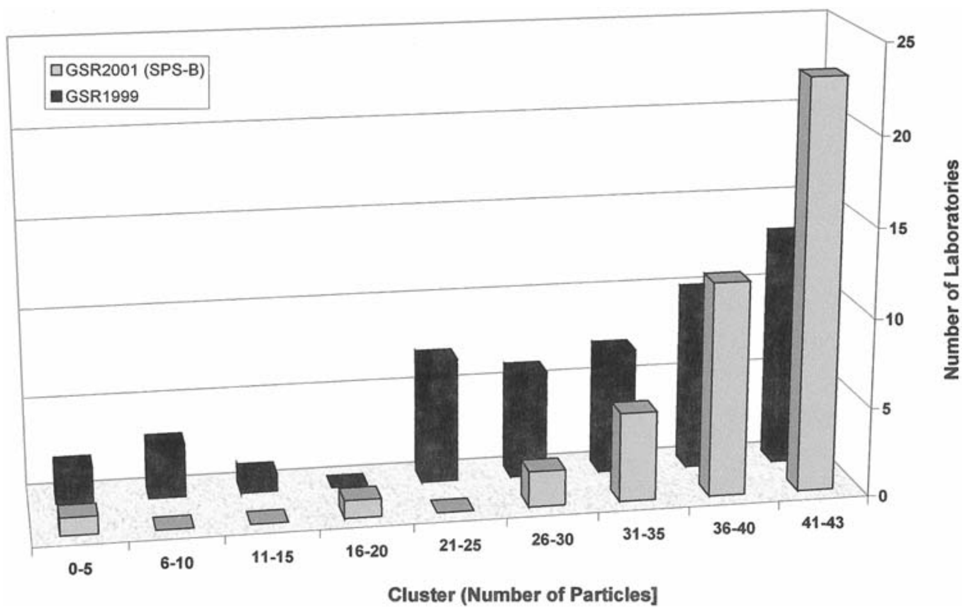

FIG. 4—*Cluster frequencies of the laboratories results.*

manufacturing companies of the instrumental equipment to take part in the optimization and subsequently improve their systems.

The sample material is also suitable for validation purposes when measurements are performed regularly and documented according to quality assurance instructions. As the sample material is long-term stable, it could fulfill the criteria of a calibration standard for GSR.

Future work is intended to carry on improving the quality of the test items in order to produce an even more authentic sample material, e.g., introducing a C-matrix instead of the actual Si-matrix, thus reducing the background Si-signal that sometimes interferes in the detection.

The application of the Youden scheme for the evaluation has proved itself to be worthwhile. It may also be used in future tests to demonstrate the significance of systematic errors.

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