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TECHNICAL NOTE

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GSR2005—Continuity of the ENFSI Proficiency Test 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 energy-dispersive scanning electron microanalysis (SEM/EDX) is carried out in a 2 years term. The latest test was performed in 2005/2006 and was denoted as *GSR2005*. Seventy-five laboratories from 28 countries participated in this proficiency test and submitted in total 83 independent data-sets. The participating laboratories were requested to determine the total number of PbSbBa containing particles on a synthetic test sample following their own laboratory specific methods of automated GSR particle search and detection by SEM/EDX. Furthermore size and position of the detected particles had to be reported by the laboratories and were evaluated statistically. The results were compiled by means of *z*-scores according to the IUPAC and EURACHEM guidelines—assessing individual laboratory achievements (inter-laboratory) as well as intra-laboratory performance—and were compared to the results of the previous proficiency test run *GSR2003* (1). The comparison shows that there is a noticeable improvement in the method's detection capability.

KEYWORDS: forensic science, gunshot residues, scanning electron microscopy, GSR, SEM/EDX, ENFSI, proficiency testing, *z*-scores

The ENFSI proficiency test program on identification of GSR (gunshot residues) is an ongoing proficiency test organized by the ENFSI expert working group "Firearms". The history of these tests, *GSR2001* and *GSR2003,* can be read in previous reports (1,2). The aim of the GSR proficiency tests is not a competition between the participating laboratories. The aim is to detect deficiencies in instrumental settings, hardware and software used, deficiencies which result in either missing particles or multiple detection of particles. The participating laboratories are asked to use their standard operating procedures for the detection of GSR particles by scanning electron microscopy equipped with X-ray microanalysis (SEM/EDX).

Materials and Methods

The test items for the GSR2005 proficiency test consisted of specially prepared, identical samples in accordance with the ISO 5725 for the performance of proficiency tests (3).

The samples were prepared artificially (1) and consisted of a number of particles with the composition characteristic for real GSR—namely of lead (Pb), antimony (Sb), and barium (Ba). As in previous tests, submicron particles have been included in order to evaluate the capability of some laboratories in detecting submicron particles using their standard parameter settings in routine analysis. Each of the samples contained in total 100 particles in four different size classes: 0.5, 0.8, 1.2, and 2.4 μm in diameter. For further statistical evaluations only the number of particles per size category was slightly changed but remained comparable to that used in the previous test (GSR2003) (1). Three different layouts were applied within the sample production, where the location of the 100 PbSbBa-particles was varied. Sets of identical samples containing synthetic GSR particles were dispatched to the participating laboratories. The number, the size, and the location of the particles had to be reported to the organizers within a limited time period.

Test samples were sent to 81 laboratories, which had accepted the conditions of participation (Table 1). This number is remarkably higher than the 56 laboratories in the previous test (1). Not only European laboratories, but also 25 non-European laboratories participated in this test. Altogether 83 data sets from 75 laboratories were considered in the statistical evaluation of the test (some laboratories submitted more than one result for different SEM/EDX systems).

After the test, the participating laboratories received a complete evaluation of their own results in terms of finding and/or missing particles. In this way, the participants are given the opportunity to comment on their performance assessment. Additionally, laboratories with poor results may question their settings and procedures and, as a consequence, improve them.

Laboratory evaluation and data assessment were performed in the same way as reported in the previous studies (1) according to ISO 13528 (4). 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 (5,6). Therefore the robust statistical method of "Q/Hampel" was used which meets the standard DIN 38402 A45 (= ISO/DIS 20612) (7,8). The values of all characteristics used for the calculation of the *z*-scores are shown in Table 2. The evaluated data of all participants are summarized in Table 3.

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TABLE 1—*List of participating laboratories (GSR2005).*

Participating Laboratories (Laboratories' Names—as Submitted—Sorted Alphabetically per Country)		
Forensic Science SA	Adelaide	Australia
New South Wales Police, Forensic Microanalysis Laboratory	Broadway	Australia
Queensland Police Service Scientific Section, Analytical Services Unit*	Brisbane	Australia
Bundeskriminalamt, Abteilung 6.2.4.	Vienna	Austria
Nationaal Instituut voor Criminalistiek en Criminologie	Brussels	Belgium
Laboratorio de Microscopia Electronica, Universidade Federal do Rio de Janeiro	Rio de Janeiro	Brazil
Centre of Forensic Sciences	Toronto	Canada
Royal Canadian Mounted Police	Ottawa	Canada
Forensic Science Center	Zagreb	Croatia
Institute of Criminalistics Prague	Prague	Czech Republic
Danish Technological Institute	Taastrup	Denmark
Estonian Police Forensic, Service Centre	Tallinn	Estonia
National Bureau of Investigation	Vantaa	Finland
Forensics Sciences Laboratory of Marseille, Department of Physico-Chemistry	Marseille	France
Institut de recherche criminelle de la Gendarmerie Nationale	Rosny-sous-Bois	France
Laboratoire de Police Scientifique	Toulouse	France
Laboratoire de Police Scientifique	Paris	France
Laboratoire de Police Scientifique de Lille	Lille	France
Laboratoire de Police Scientifique Lyon	Lyon	France
Bayerisches Landeskriminalamt	Munich	Germany
Bundeskriminalamt; KT23, SEM/EDX-system No. 1	Wiesbaden	Germany
Bundeskriminalamt; KT23, SEM/EDX-system No. 2	Wiesbaden	Germany
Hessisches Landeskriminalamt—KTI	Wiesbaden	Germany
KTI / LKA Baden-Württemberg	Stuttgart	Germany
Landeskriminalamt Brandenburg, Dezernat Forensische Chemie	Basdorf	Germany
Landeskriminalamt Sachsen-Anhalt	Magdeburg	Germany
LKA Berlin, Kompetenzzentrum Kriminaltechnik, KT 44	Berlin	Germany
LKA Mecklenburg—Vorpommern, Abteilung 5	Rampe	Germany
LKA Niedersachsen	Hannover	Germany
LKA Nordrhein—Westfalen	Düsseldorf	Germany
LKA Rheinland-Pfalz	Mainz	Germany
LKA Sachsen	Dresden	Germany
LKA Schleswig—Holstein	Kiel	Germany
LKA Thüringen	Erfurt	Germany
Polizei Hamburg, LKA 33	Hamburg	Germany
Forensic Alliance	Tamworth	Great Britain
Forensic Science Laboratory	Glasgow	Great Britain
Forensic Science Service, SEM/EDX-system No. 1	Birmingham	Great Britain
Forensic Science Service, SEM/EDX-system No. 2	Birmingham	Great Britain
Forensic Science Laboratory, Dublin	Dublin	Ireland
Div. of Identification and Forensic Science (DIFS)/Toolmarks and Materials Lab	Jerusalem	Israel
Laboratorio di Scienze Criminalistiche Universita di Torino	Turin	Italy
Reparto Carabinieri Investigazioni Scientifiche di Messina—Sezione di Balistica*	Messina	Italy
Reparto Carabinieri Investigazioni Scientifiche di Parma—Sezione di Balistica	Parma	Italy
Reparto CC Investigazioni Scientifiche—Roma	Rome	Italy
Netherlands Forensic Institute (NFI), SEM/EDX-system No. 1	The Hague	Netherlands
Netherlands Forensic Institute (NFI), SEM/EDX-system No. 2	The Hague	Netherlands
Forensic Science Northern Ireland	Belfast	Northern Ireland
National Criminal Investigation Service	Oslo	Norway
Central Forensic Laboratory of the Polish Police	Warsaw	Poland
Institute of Forensic Research	Cracow	Poland
Laboratorio de Policia Cientifica—Policia Judiciaria	Lisboa	Portugal
NIFE	Bucharest	Romania
Kriminalisticky a expertizny ustav Policajneho zboru	Bratislava	Slovakia
Ministry of Interior	Ljubljana	Slovenia
South African Police Service*	Pretoria	South Africa
Comisaría General de Policía Científica	Madrid	Spain
Guardia Civil - Servicio de Criminalistica	Madrid	Spain
INT CCFF	Madrid	Spain
National Laboratory of Forensic Science (SKL)	Linkoeping	Sweden
Forensische Chemie + Technologie	St. Gallen	Switzerland
Scientific Forensic Service of the City Police of Zurich	Zurich	Switzerland
Ministry of Interior, Gendarmerie General Command, Criminal Department*	Ankara	Turkey
Alameda County Sheriff's Office—Criminalistics Laboratory	San Leandro, CA	USA
Allegheny County Coroner's Office, Forensic Laboratory	Pittsburgh, PA	USA
Bexar County Criminal Investigation Laboratory	San Antonio, TX	USA
Colorado Bureau of Investigation	Denver, CO	USA
CT Forensic Science Laboratory*	Meriden, CT	USA
FBI Laboratory	Quantico, VA	USA
Hamilton County, Coroner's Laboratory	Cincinnati, OH	USA
Harris County Medical Examiners Office*	Houston, TX	USA

TABLE 1—*(continued)*

Participating Laboratories (Laboratories' Names—as Submitted—Sorted Alphabetically per Country)		
Honolulu Police Department, Scientific Investigation Section	Honolulu, HI	USA
Illinois State Police Forensic Science Center at Chicago	Chicago, IL	USA
Los Angeles County, Department of Coroner	Los Angeles, CA	USA
Missouri State Highway Patrol, Crime Lab	Jefferson City, MO	USA
Montana Forensic Science Division	Missoula, MO	USA
Ohio Bureau of Criminal Identification & Investigation	London, OH	USA
Orange County Sheriff-Coroner Department	Santa Ana, CA	USA
Sacramento County District Attorney, Laboratory of Forensic Services	Sacramento, CA	USA
Santa Clara County DA's Crime Lab	San Jose, CA	USA
Virginia Department of Forensic Science	Richmond, VA	USA

*These laboratories did not submit their data within the deadline of the study, and are therefore not included in the final statistical evaluation.

TABLE 2—*Mean values and standard deviations for the six different particle size categories.*

Particle Size Category (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)	56	55	3.1	5.5	3.1
TOTAL ≥ 0.8 (no. of det. PbSbBa particles with a diameter of 0.8 μm, 1.2 μm, and 2.4 μm)	86	85	6.7	8.5	6.7
SIZE 2.4 (no. of det. PbSbBa particles with a diameter of 2.4 μm)	24	23	1.1	2.3	1.1
SIZE 1.2 (no. of det. PbSbBa particles with a diameter of 1.2 μm)	32	31	3.0	3.1	3.0
SIZE 0.8 (no. of det. PbSbBa particles with a diameter of 0.8 μm)	30	29	3.8	2.9	2.9
SIZE 0.5 (no. of det. PbSbBa particles with a diameter of 0.5 μm)	14	13	4.6	1.3	1.3

*Even for the characteristics TOTAL ≥ 1.2 and TOTAL ≥ 0.8 only one missing particle in total is accepted as ′reference X′ (calculated from the test of homogeneity and stability).

TABLE 3—*Overall proficiency values for the six size categories. A comparison between GSR2005 and GSR2003.*

	Satisfactory ($|z| < 2$)	Questionable ($2 \le |z| \le 3$)	Unsatisfactory ($|z| > 3$)	Total No. of Labs Considered per Size Class
GSR2005				
TOTAL ≥ 1.2	79%	4%	17%	81
TOTAL ≥ 0.8	73%	4%	23%	71
SIZE 2.4 μm	90%	4%	6%	83
SIZE 1.2 μm	80%	5%	15%	81
SIZE 0.8 μm	69%	7%	24%	71
SIZE 0.5 μm	43%	6%	51%	61
GSR2003				
TOTAL ≥ 1.2	75%	4%	21%	56
TOTAL ≥ 0.8	67%	7%	26%	42
SIZE 2.4 μm	86%	7%	7%	56
SIZE 1.2 μm	70%	2%	28%	56
SIZE 0.8 μm	45%	17%	38%	42
SIZE 0.5 μm	26%	6%	68%	35

In the following, the distribution of z-scores for each particle class and the two characteristics *TOTAL* ≥ 0.8 and *TOTAL* ≥ 1.2 are shown as histograms. For example, almost 30% of the 61 laboratories that analyzed 0.5 μm particles exhibit even a z-score of 0 and about 43% exhibit a still satisfactory z-score between −2 and 0. The exact percentages can also be referred to in Table 3.

For satisfactory results, the absolute value of the z-score should be less than 2. The values $2 \le |z| \le 3$ are questionable and $|z| > 3$ unsatisfactory (4). Some laboratories are not interested in the detection of submicron particles with their standard instrumental settings, whereas others are supposed to detect even 0.5 μm particles. This situation was taken into consideration when calculating the percentage of laboratories and the z-scores for different particle sizes. For comparison, the results of the previous test are also shown in Table 3. It can be seen in this table that the number of laboratories with "positive" results ($|z| < 2$) has increased, particularly for the detection of submicron particles.

Table 3 shows the *GSR2005* laboratory assessments compared to those obtained in the previous proficiency test *GSR2003*. The results show that there is a slight improvement in the performance of the participating laboratories in terms of detecting particles bigger than 0.8 μm. Furthermore, the detection capabilities of the particle size categories 0.5 μm and 0.8 μm show that the laboratories did optimize their measuring parameters in such a way that smaller particles can be detected with a higher possibility. Those particles generally tend to be missed due to instrumental limitations.

Additionally, the design of the proficiency test studies allows a comparison of the obtained achievements for those laboratories that participated in both, the *GSR2005* and *GSR2003*, tests in Fig. 1. Therefore a certain feature (e.g., a distinct particle size class) is chosen and the performances within both proficiency tests are

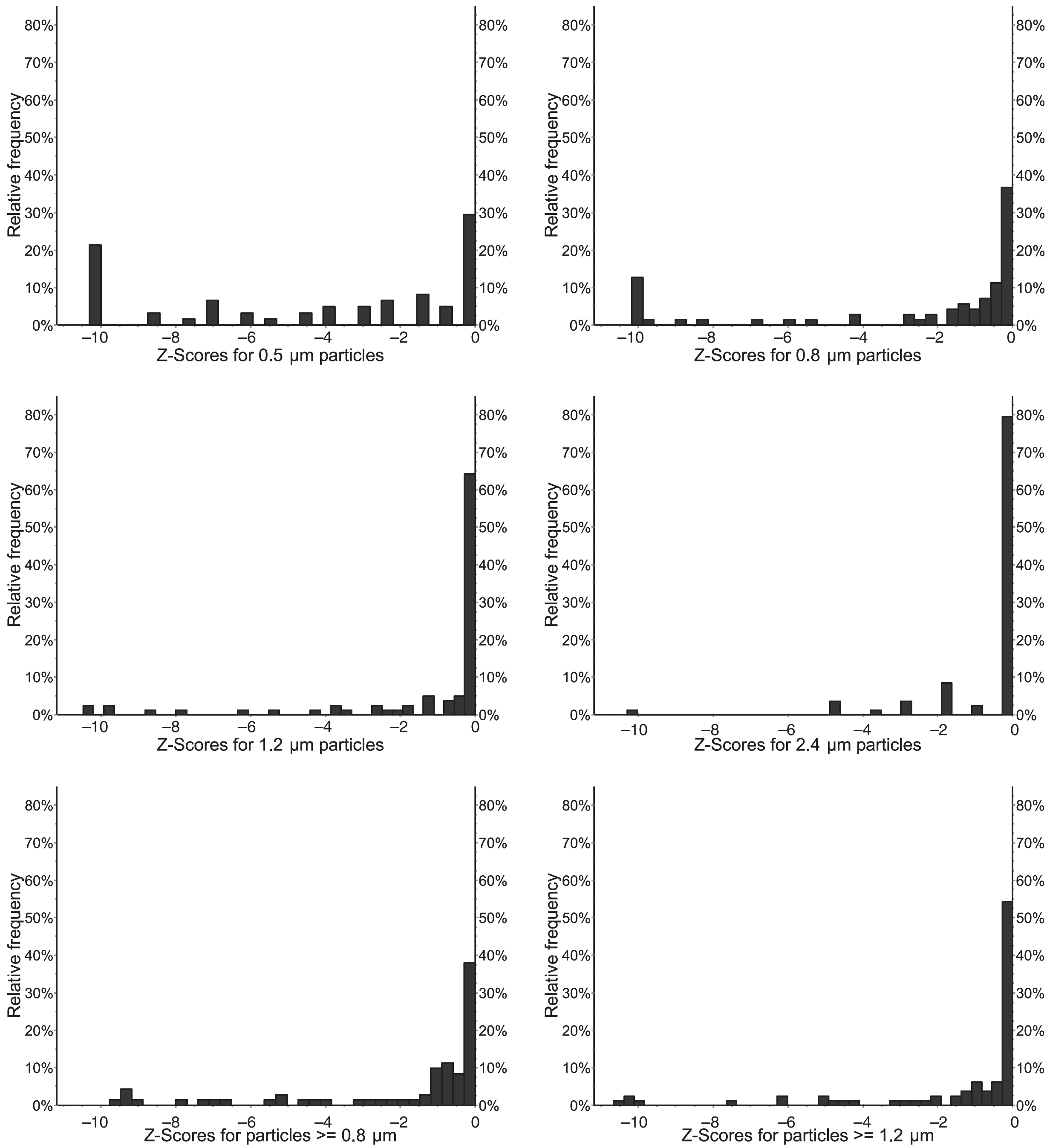

FIG. 1—*Histograms of z-scores for the different size categories; based on the number of valid laboratories (see Table 3).*

shown for the given laboratories (evaluation according to Youden (9)). Again an improvement can be ascertained, which is also illustrated in Fig. 2, where the participating laboratories are represented by their Lab-Codes. This figure shows the correlation between the *GSR2003* and *GSR2005* results for the *z*-score evaluation of the 0.8 μm particles; it is based on 38 laboratories. It has to be noted that several laboratories (illustrated by small squares) do overlap each other and may not be visible.

Those laboratories clustered within the $|z| < 2$ area in the top right corner (40%) performed satisfactory in both tests. Laboratories which are found in the light-grey area (39%) showed an improved performance in *GSR2005*. Laboratories located in the dark-grey area (3%) performed well in 2003 but show a change for the worse in 2005. Eighteen percent of the laboratories did not perform satisfactory in either proficiency tests, *GSR2003* and *GSR2005,* respectively. However, one of them showed an improvement and is within the "questionable" range in *GSR2005*. Laboratories located close to the black diagonal show reproducible but continuously non-satisfactory results.

Figure 3 shows the Youden evaluation for the 2.4 μm particles based on 51 laboratories. It illustrates that almost all laboratories (80%) performed successfully within this size category in both proficiency tests, *GSR2003* and *GSR2005*. Fourteen percent of the laboratories showed an improved performance in *GSR2005,* 4% performed well in 2003 but show a change for the worse in 2005 and only one laboratory (2%) did not perform satisfactory in any

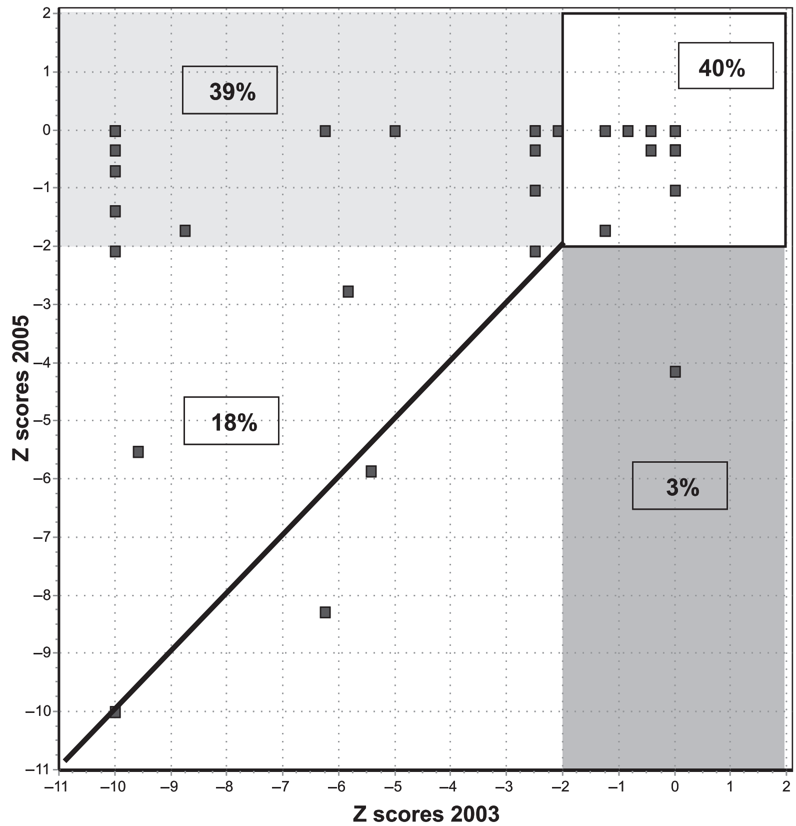

FIG. 2—*Youdenplot of z-scores for particle size 0.8 μm; based on 38 laboratories.*

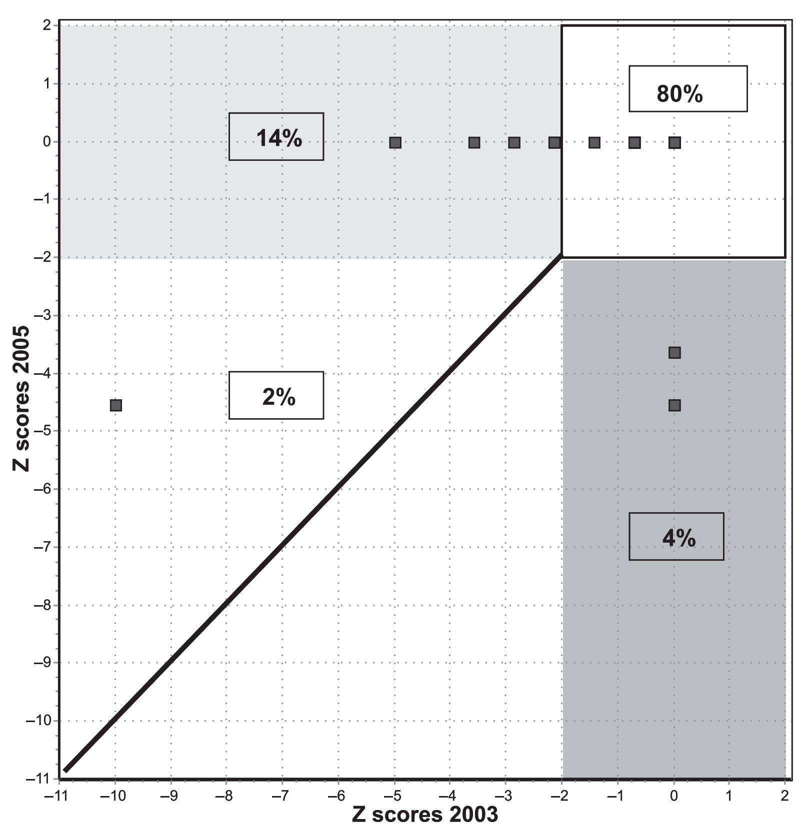

FIG. 3—*Youdenplot of z-scores for particle size 2.4 μm; based on 51 laboratories.*

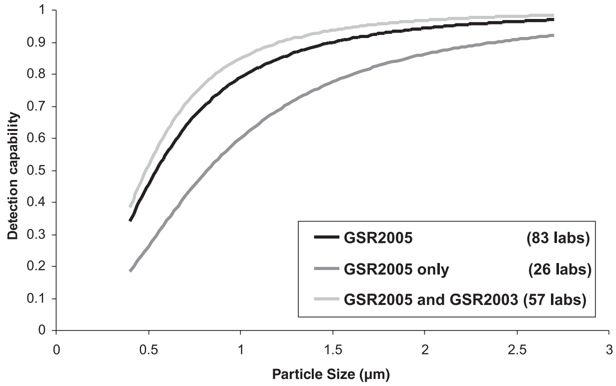

FIG. 4—*Method's detection capability, calculated from the results of the GSR2005 test.*

proficiency test. However, also for this laboratory an improvement is evident.

To estimate the overall quality of the SEM/EDX method in GSR investigation, the method's detection capability was determined (1). It describes the probability for a randomly selected laboratory to detect a particle of a certain particle size. Figure 4 illustrates the overall detection capability of the SEM/EDX method, calculated from the data of the *GSR2005* test. A detailed description on the statistical evaluation of this function is given in (1) and (10). According to this figure, a randomly selected laboratory will detect in average a 1 μm particle with a probability of approximately 79% and a 1.5 μm particle with a probability of 90%.

The figure also reveals that an even better detection capability is obtained for laboratories that participated in both tests, *GSR2003* and *GSR2005*. For this case a randomly selected laboratory will detect in average a 1 μm particle with a probability of approximately 85% and a 1.5 μm particle with a probability of 94%. For laboratories that participated in *GSR2005* for the first time, the detection capability is less convincing: a randomly selected laboratory will detect in average a 1 μm particle with a probability of only 60% and a 1.5 μm particle with a probability of 78%.

Conclusions

As the proficiency test *GSR2005* was intentionally designed in the same manner as the *GSR2003* test, a direct comparison of the assessments is permissible.

The laboratories that participated in 2003 and 2005 show a noticeable improvement in the performance of the *GSR2005* test (see Fig. 4). In contrast to this, laboratories that took part in the *GSR2005* test for the first time (and which can be considered as a "control group") entirely show a lower detection capability rate. It can be deduced from the evaluations that a continuous participation in the proficiency test program contributes to an improvement in the performance of the individual SEM/EDX systems, in particular for the detection of submicron GSR particles. This allows the laboratories to react to deficits in the performance of their systems based on the individual detection capabilities, and thus gives the opportunity for system optimization and validation, especially as the test sample remains with the laboratories after the test for further use.

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