Forensic PT

The ENFSI Proficiency Testing Programme on Identification of GSR by SEM/EDX

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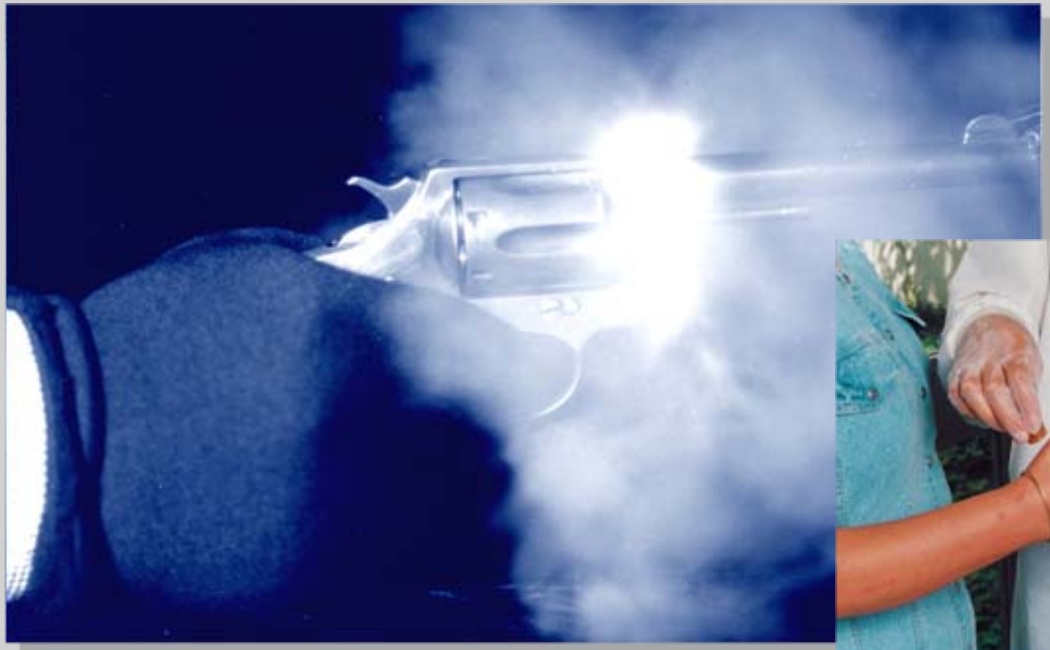
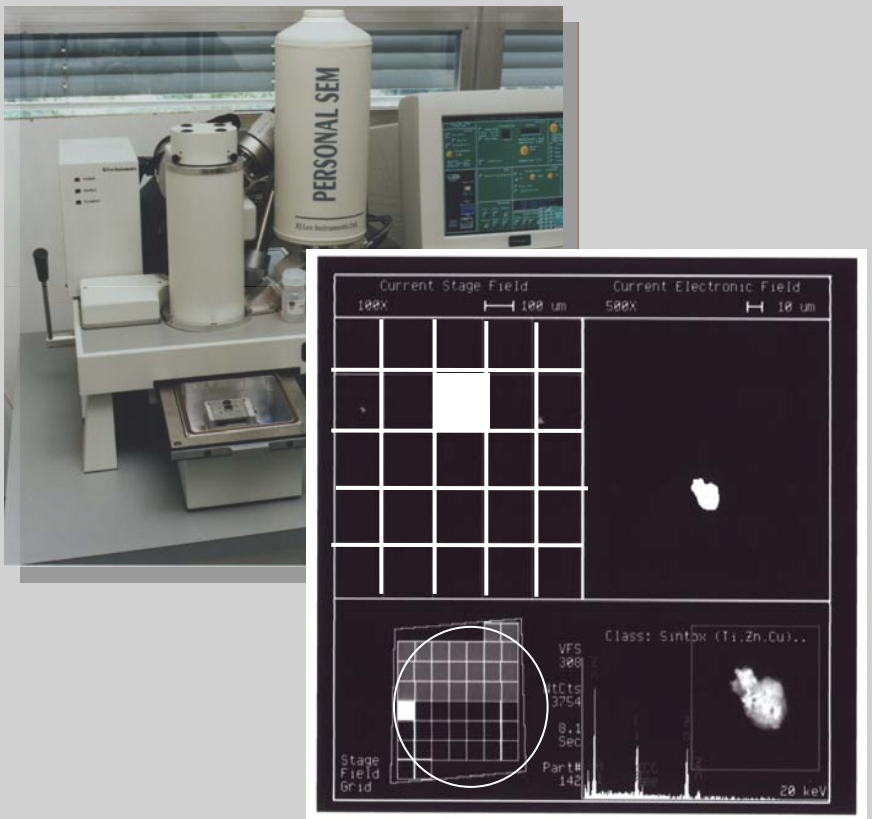
A G E N D A

- What are Gunshot Residues (GSR)
- PT Design / Sample Production
- Data Evaluation
- Inter - Laboratory Applications
  - Proficiency Testing / Method Validation
- In - Laboratory Applications
  - QA / System Validation
- Outlook / Future Applications

Sampling of GSR

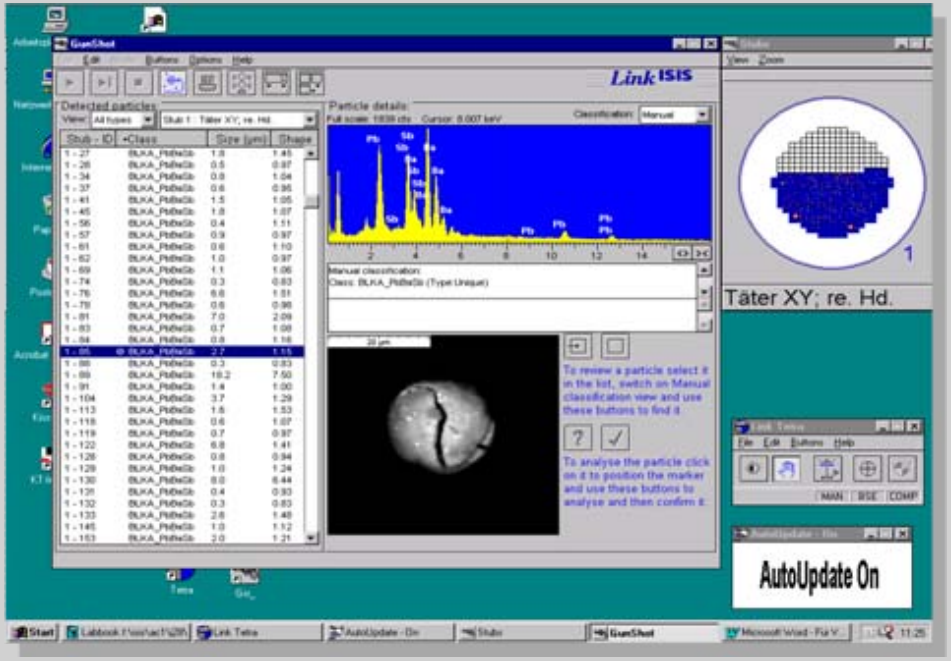


Detection of GSR by SEM/EDX



- Automated particle search by using compositional contrast (BSE), imaging, acquisition of EDX-spectrum
- classification of particles according to their chemical composition
- manual verification of GSR indicative particles (EDX-spectrum & morphology)

Detection of GSR by SEM/EDX



- Automated particle search by using compositional contrast (BSE), imaging, acquisition of EDX-spectrum
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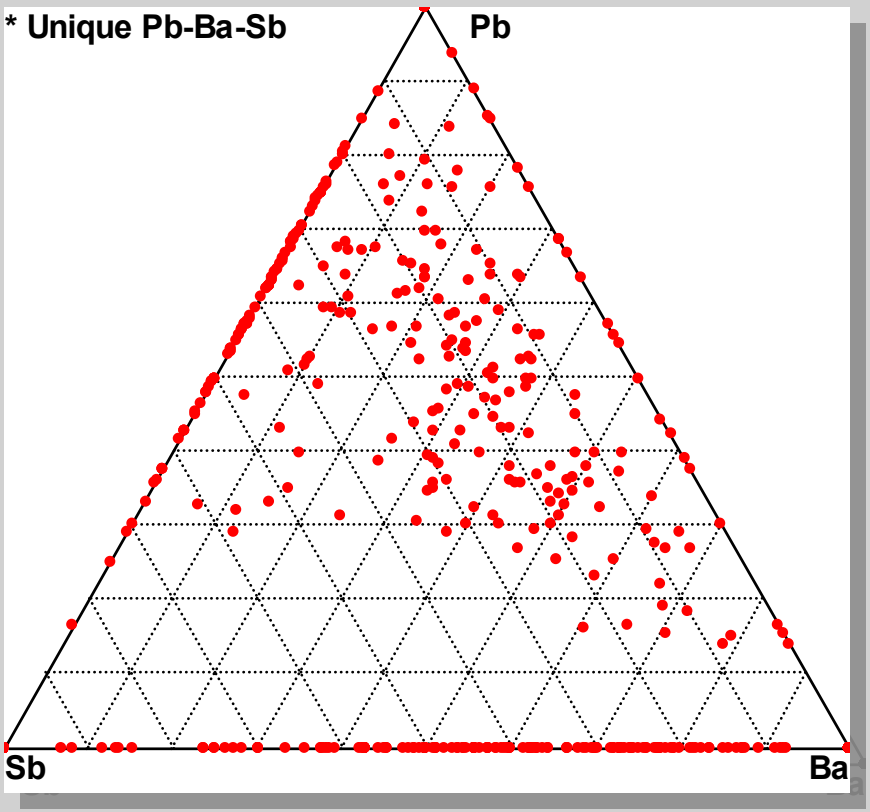
Ternary Diagram



Problems in Automated Particle Analysis

Automated SEM/EDX systems for particle detection

Is there a need for a standard?

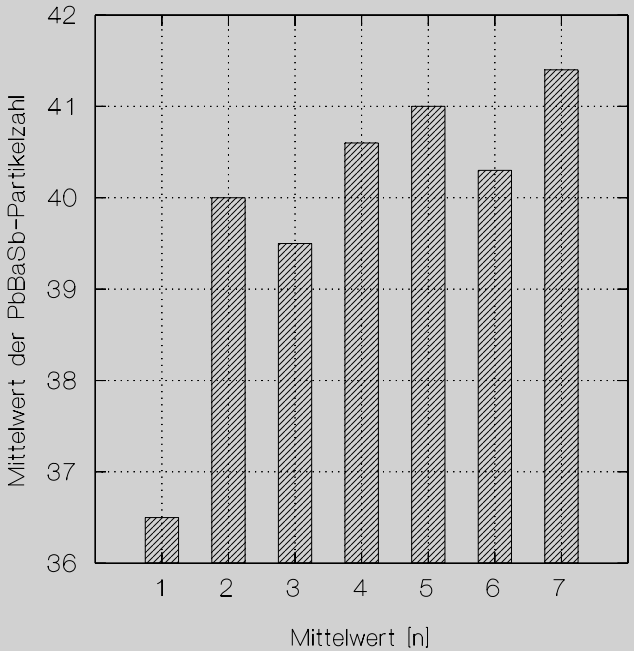
system checking for:

- reproducibility
- reliability

System Validation

Question: Will all particles be detected?

⇒ **test on the robustness of mean**



Conclusions:

⇒ **5 measurements necessary**
(with a stat. certainty of 95%)

SYSTEMATIC ERRORS

Demands on a particle standard

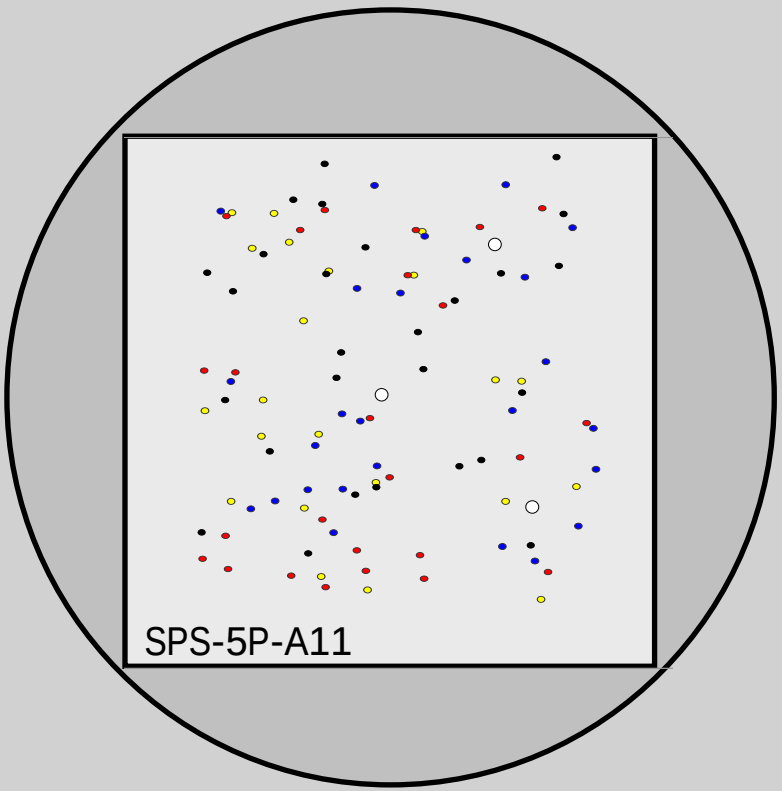
A sample with:

- known number of GSR particles
- known chemical compositions
- known particle sizes
- known location of the particles

Preparing an "artificial" GSR sample

➔ Silicon chip

Sample design



- 1/2 “ stub
- 8x8mm Si chip
- 0.5 µm: 22 particles
- 0.8 µm: 25 particles
- 1.2 µm: 26 particles
- 2.4 µm: 27 particles

O 10 µm: 3 particles

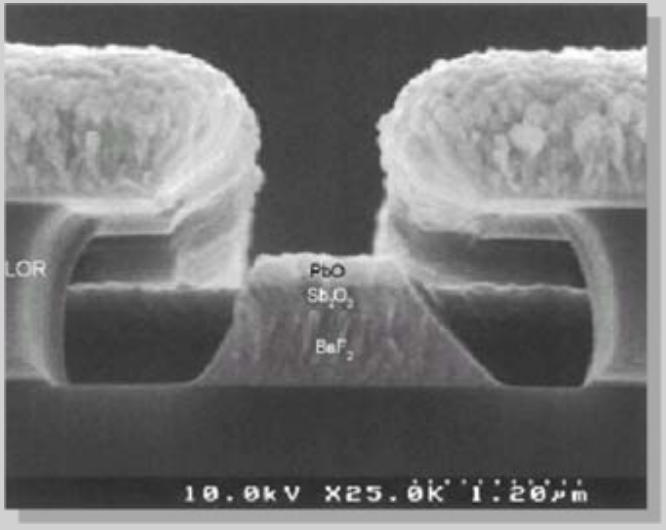
- unique sample ID

patent no: DE 199 32 357 C2

Sample production

- 4" wafer
- 8x8 mm² chips
- unique sample ID

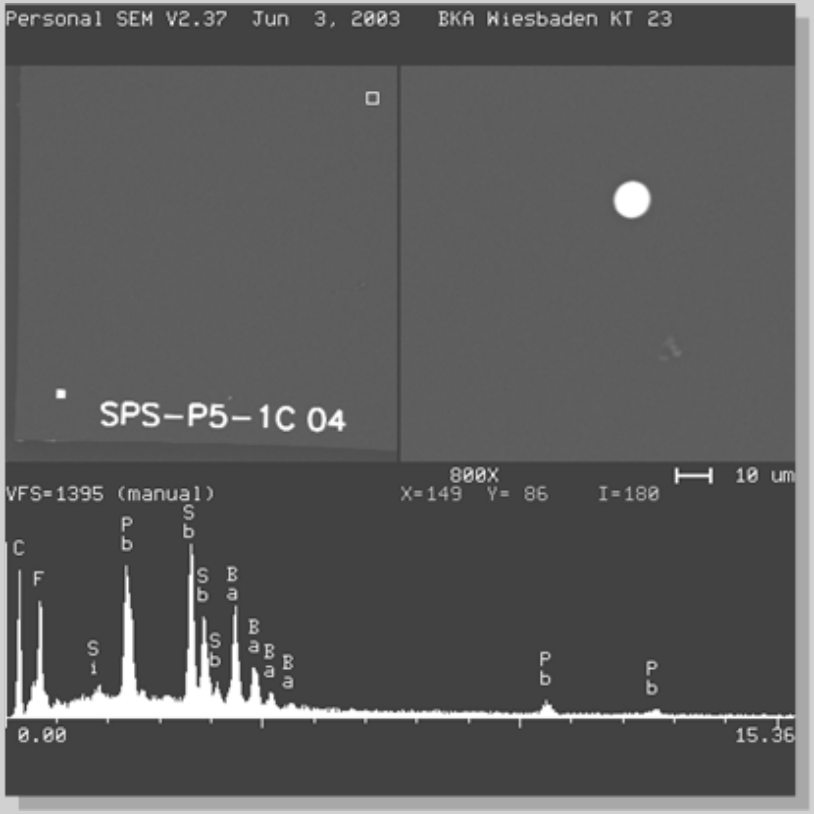
Sample production



Synthetic GSR-particle
before Lift-Off-Process

Mounting of chip on SEM-stub

Sample production



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Application of the synthetic particle standard in PT

- Important features of the GSR-standard as a PT material:
  - Defined number, size, position of the particles
  - Defined chemical composition
  - High sample stability
  - Can be examined in the same way as samples form real cases.

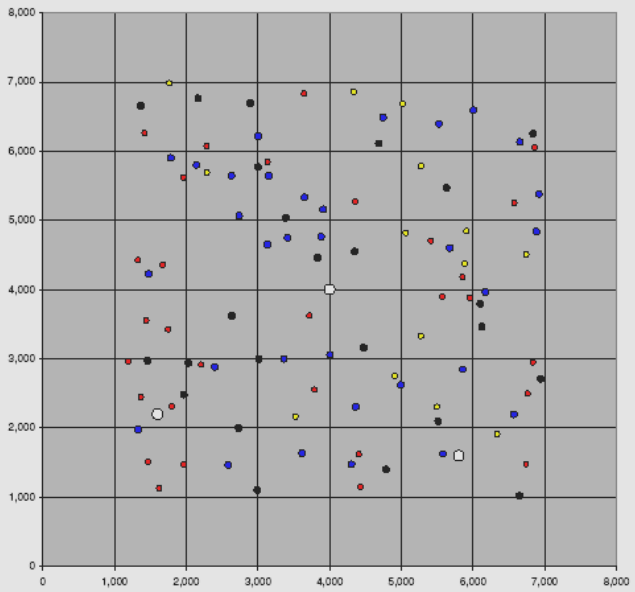
History of the GSR PT Programme

- 1995 ENFSI: first discussions within the EWG "Firearms"
- 1996 first study ("collaborative exercise")
- 1999 “proficiency test” GSR1999 (study)
  ➔ first attempt with synthetic GSR, PbSb-particles
- 2001 1st proficiency test GSR2001
  ➔ PbSbBa-particles
- 2003 “GSR2003”; Final Meeting in Bad Camberg, Germany
- 2005 “GSR2005”; Final Meeting in Copenhagen; Denmark
- 2008 “GSR2008”; Final Meeting in Dubrovnik, Croatia

Data evaluation

- Export of particle coordinates to Excel® (PbBaSb, PbBa, PbSb, BaSb)
- Comparison with sample layout (Master)
  1. Manually by printout of Excel® data (XY-plot) and comparison with template (e.g. overhead transparency)
  2. By transformation of the particle coordinates into the sample template in Excel®
- In both cases the 10 µm particles are used as a landmark
- Checking-off of the detected particles regarding the different size categories

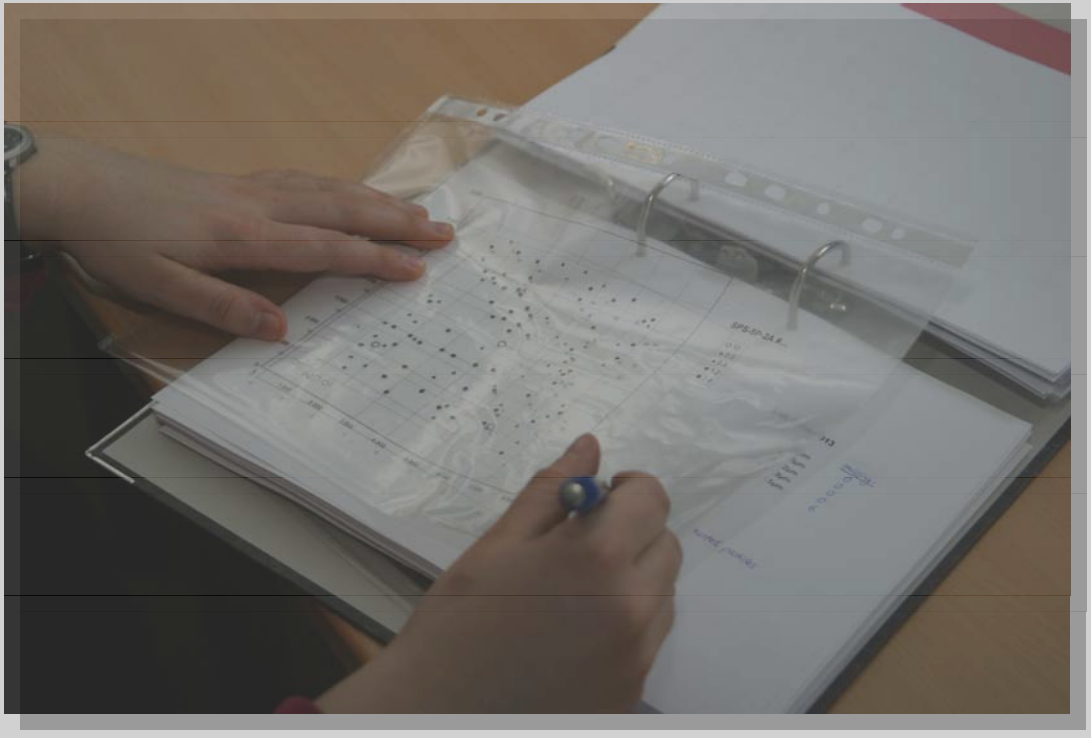
Data evaluation



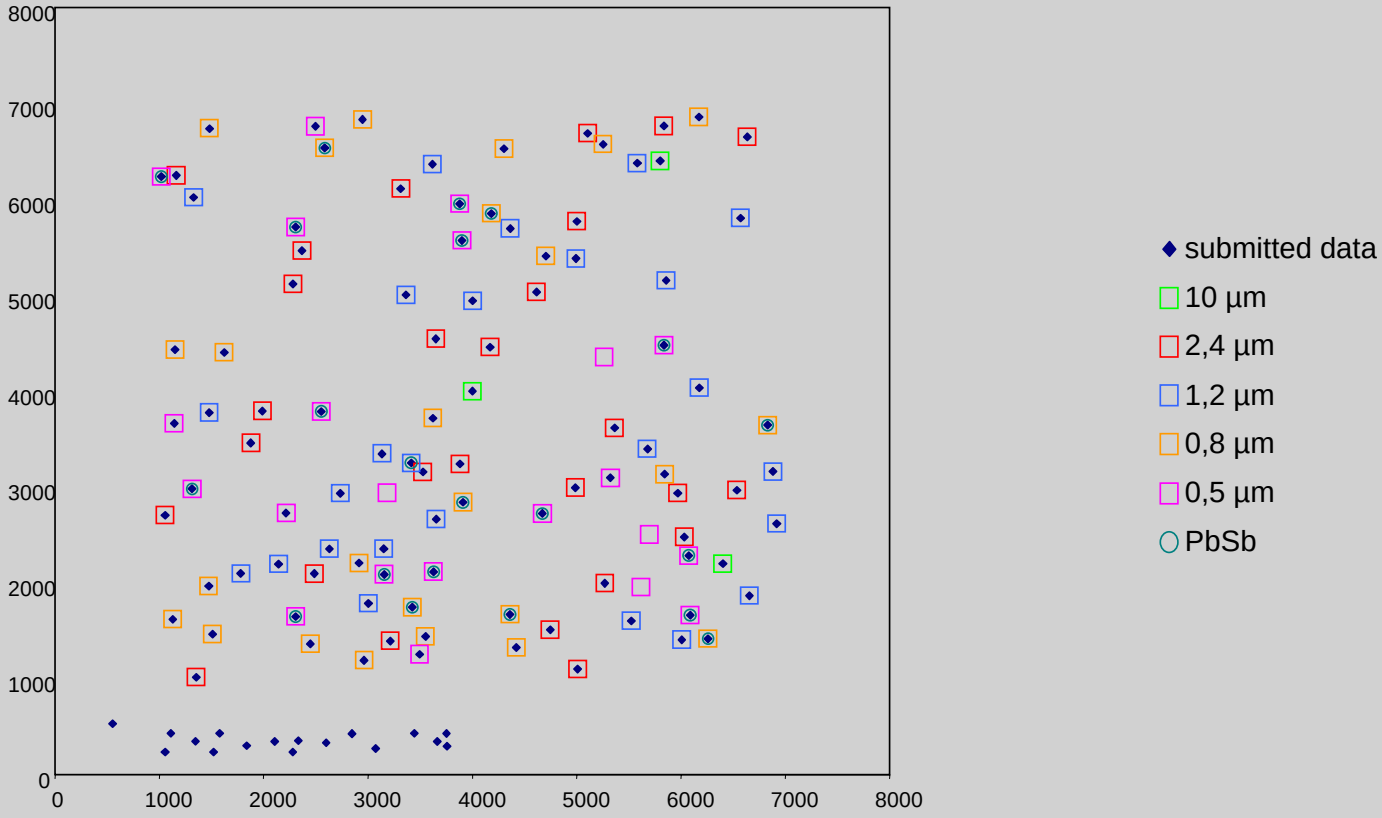
Lab-ID: #117

Feature no.#	Field no.#	PbBaSb	PbBa	PbSb	BaSb	ECD (µm)	Stage X (mm)	Stage Y (mm)
38	74	1	0	0	0	0.6	61.413	44.445
87	146	1	0	0	0	0.7	58.769	47.438
42	79	1	0	0	0	0.9	58.653	44.891
76	131	1	0	0	0	0.9	61.555	46.582
82	142	1	0	0	0	0.9	58.250	48.721
91	163	1	0	0	0	0.9	60.874	45.484
61	111	1	0	0	0	1.0	60.029	43.922
65	117	1	0	0	0	1.0	61.995	44.075
109	165	1	0	0	0	1.0	59.233	46.111
138	177	1	0	0	0	1.0	59.791	47.164
91	149	1	0	0	0	1.1	60.582	47.324
92	149	1	0	0	0	1.1	60.303	47.609
18	43	1	0	0	0	1.1	62.058	43.142
17	41	1	0	0	0	1.2	60.957	43.213
25	58	1	0	0	0	1.2	58.436	44.037
118	172	1	0	0	0	1.2	58.316	48.599
22	52	1	0	0	0	1.2	61.082	43.516
21	48	1	0	0	0	1.3	58.884	43.716
14	39	1	0	0	0	1.3	60.089	43.091
10	31	1	0	0	0	1.4	61.554	42.968
55	97	1	0	0	0	1.4	57.665	45.394
97	158	1	0	0	0	1.4	60.601	47.707
120	173	1	0	0	0	1.5	59.095	48.662
12	36	1	0	0	0	1.5	58.253	43.175
47	80	1	0	0	0	1.5	59.559	44.803
60	101	1	0	0	0	1.6	59.741	45.643
89	148	1	0	0	0	1.6	59.890	47.427
35	69	1	0	0	0	1.7	58.853	44.382
58	100	1	0	0	0	1.7	59.341	45.670
48	81	1	0	0	0	1.7	59.856	44.897

Manual evaluation



Evaluation in Excel



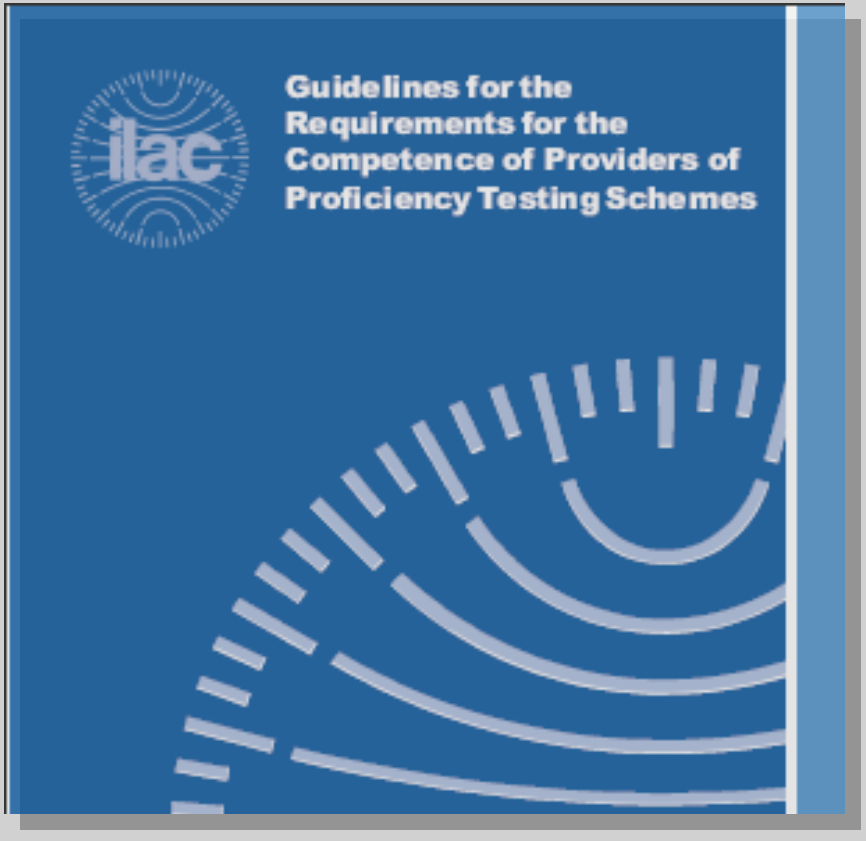
Inter- laboratory application

Samle material for proficiency testing

- laboratory assessment
- method assessment

  (e.g. particle size; detection capability)

ASCLD-LAB approval



ILAC-Guide G13-2000

ISO 5725 1 - 4

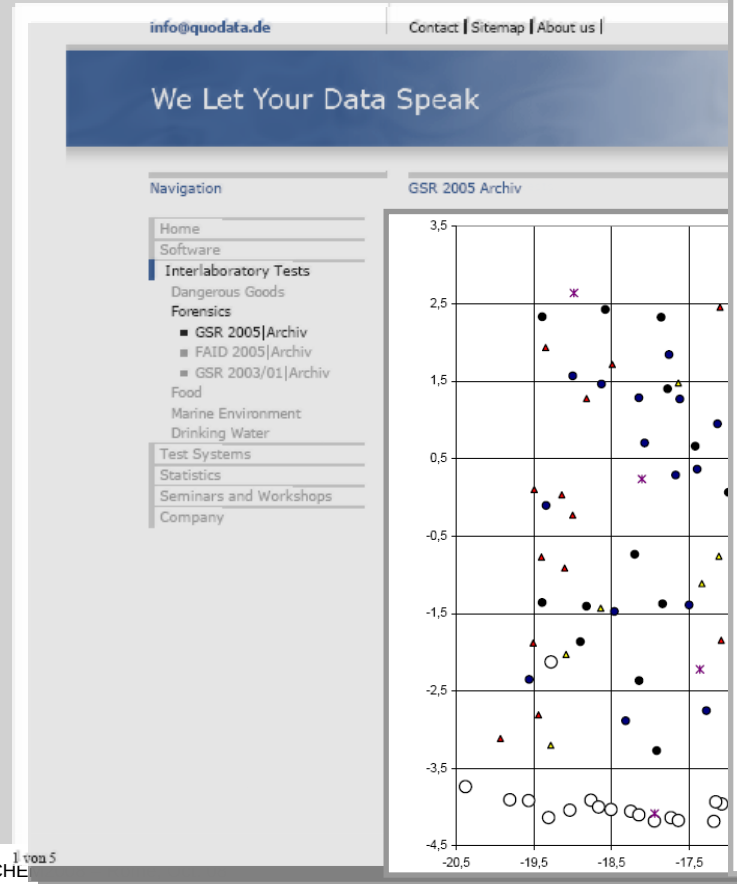
ISO Guide 43-1

GSR Proficiency Test

GSR Proficiency Testing Programme

- within ENFSI (**E**uropean **N**etwork of **F**orensic **S**cience **I**nstitutes)
- granted by EU (OISIN, AGIS program)
- GSR1999, GSR2001, GSR2003, GSR2003, GSR2005, GSR2008
- all results on **www.quodata.de**
- published in JFS (**vol. 53-1; 2008**)

www.quodata.de



Enclosure to the certificate of participation in the interlaboratory test GSR2005

for the laboratory

[redacted]

Lab Code: 117

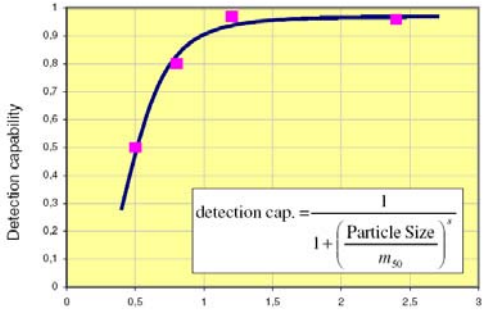
[redacted]

Description	True Value	Assigned Value*	Lab result**	s.d. used	Z - Score
Count of 0.5µm particles correctly detected	14	13	7	1,3	-4,6
Count of 0.8µm particles correctly detected	30	29	24	2,9	-1,7
Count of 1.2µm particles correctly detected	32	31	31	3,0	0,0
Count of 2.4µm particles correctly detected	24	23	23	1,1	0,0
Count of particles >=0.8µm correctly detected	86	85	78	6,7	-1,0
Count of particles >=1.2µm correctly detected	56	55	54	3,1	-0,3

*) In order to keep the conditions equal for all participants, a defect of one particle at the most over all size classes per test sample was allowed.
**) If the lab result equals the true number of particles, it is set to the assigned value (true value – 1), in order to avoid inconsistent Z scores

Detection capability versus particle size

50% - Percentile: m_{50} = 0.5 µm; 90% - Percentile: m_{90} = 1.0 µm; steepness: s = -4



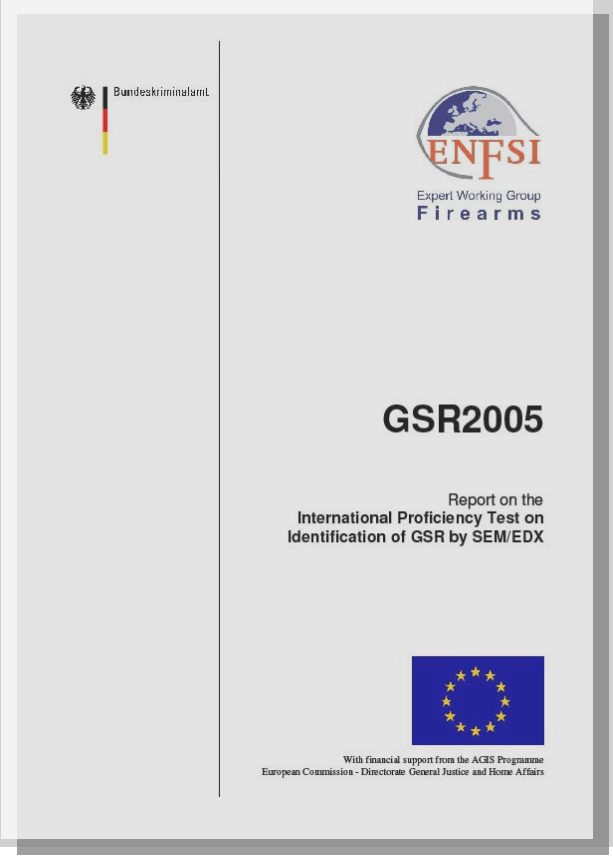
ENFSI Expert Working Group Firearms

ProLab 2006 / quo data GmbH.
http://www.quodata.de/cms-bka

quo data

1 von 5

Reports


Bundeskriminalamt
ENFSI
Expert Working Group
Firearms
GSR2005
Report on the
International Proficiency Test on
Identification of GSR by SEM/EDX
With financial support from the AGIS Programme
European Commission - Directorate General Justice and Home Affairs

Z-score Assessment

Z-scores

- According to "Internat. Harmon. Protocol for Proficiency Testing of (Chemical) Analytical Laboratories"
- Determination of mean value (M) and standard deviation (S)
- Assessment of the individual success rate by ”Z-scores"

$$Z = \frac{[\text{no. of detected part.}] - M}{S}$$

(ISO Guide 43-2; EURACHEM; ILAC-Guide G13)

Reports

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for the laboratory

Bundeskriminalamt; KT23

Lab Code: 117
Thaerstrasse 11
65193 Wiesbaden

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Count of particles >=0.8µm correctly detected	86
Count of particles >=1.2µm correctly detected	56

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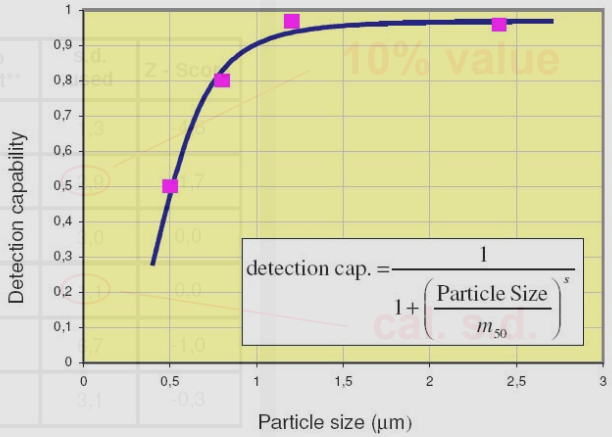

ProLab 2006 / quo data GmbH.
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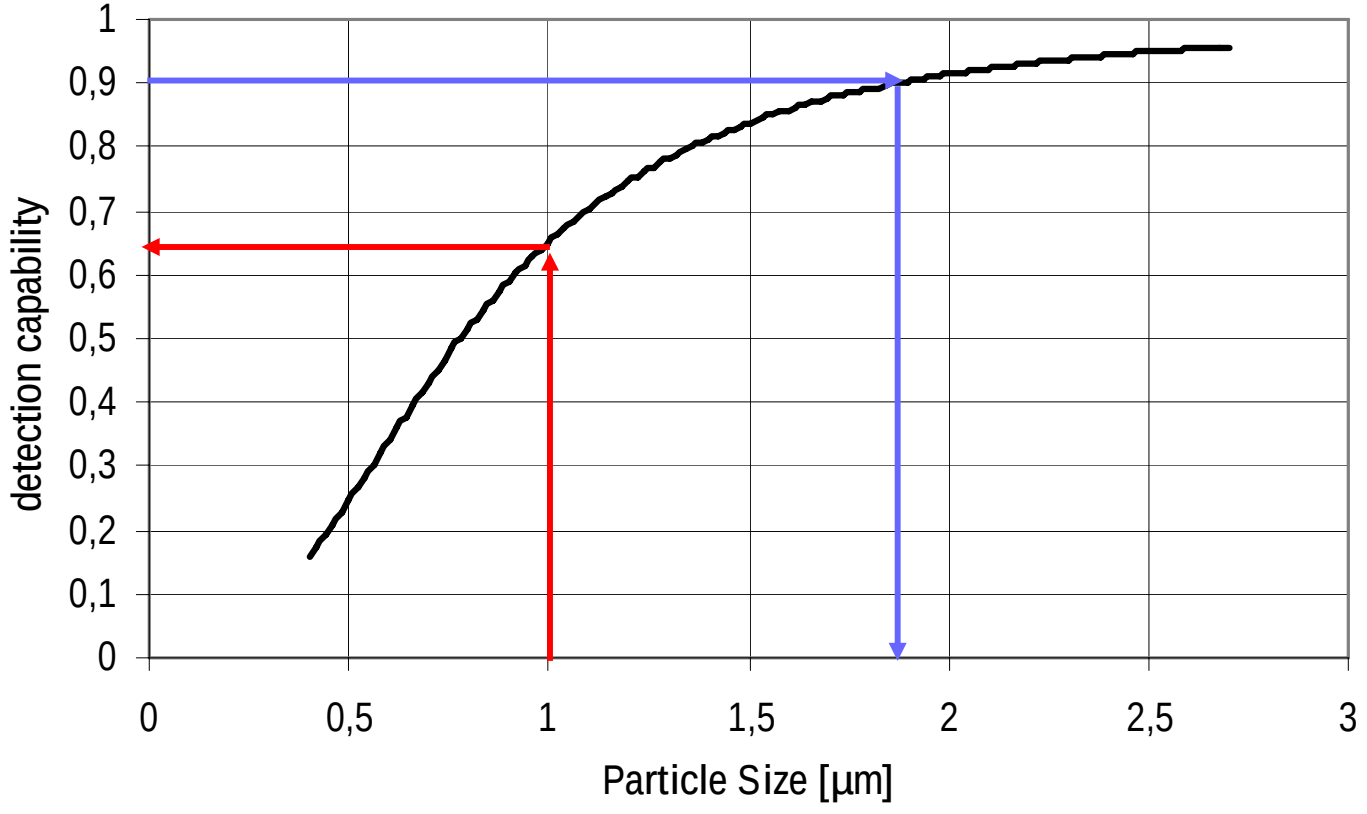
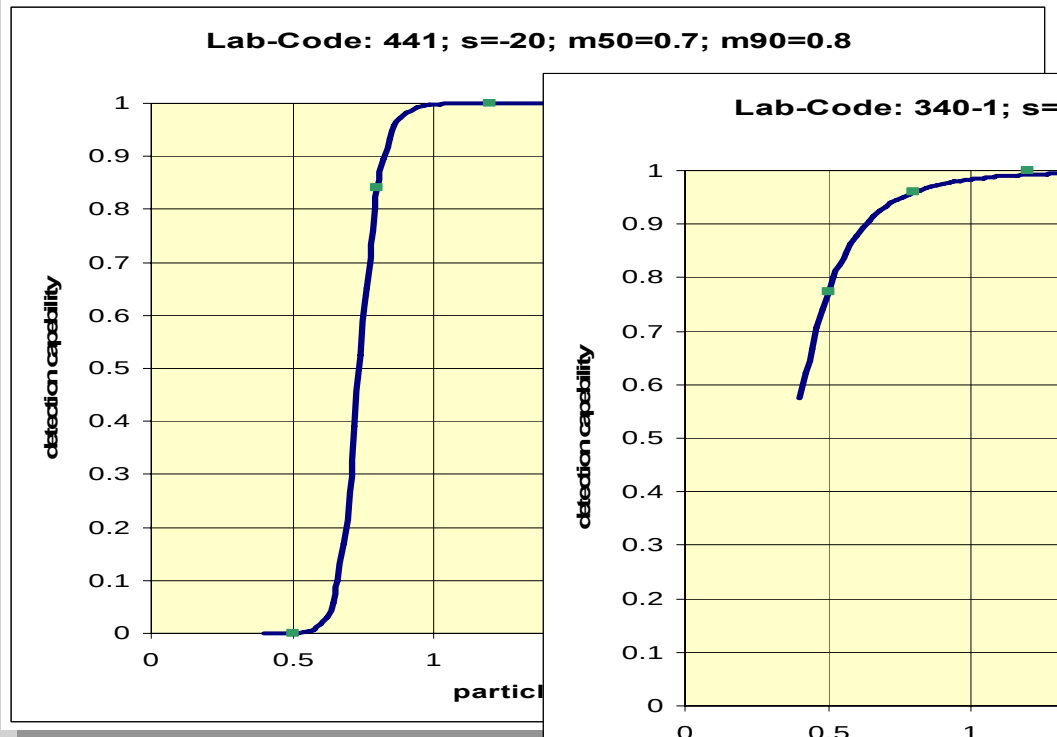
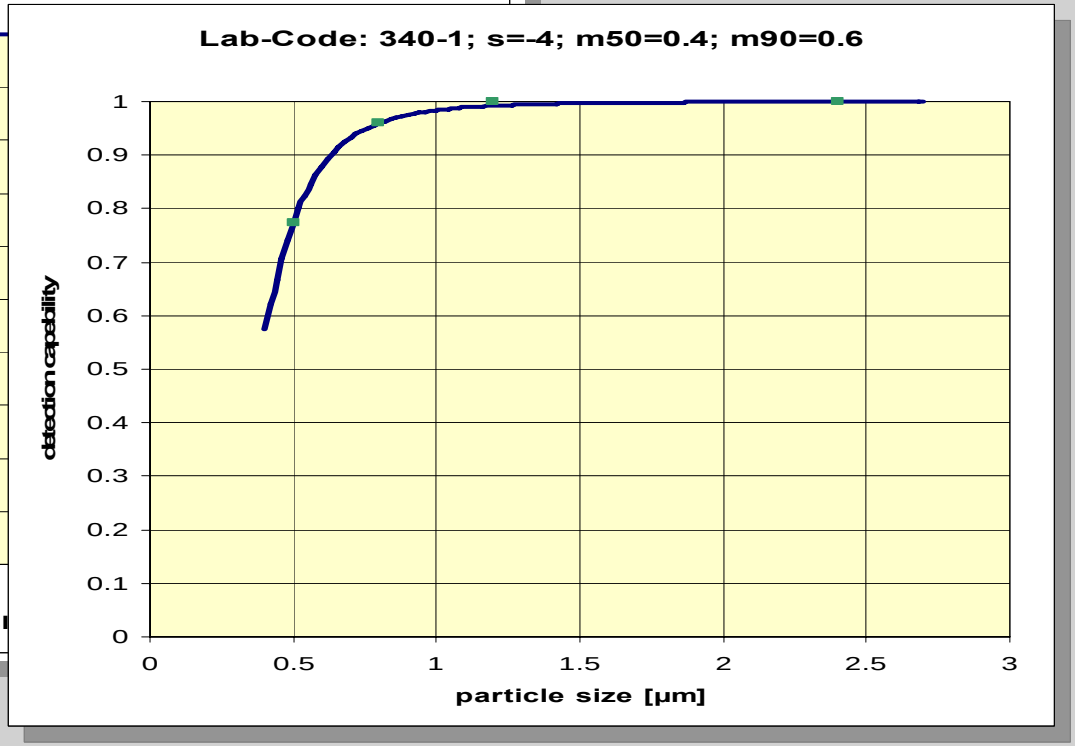
ENFSI Expert Working Group Firearms

Detection Capability

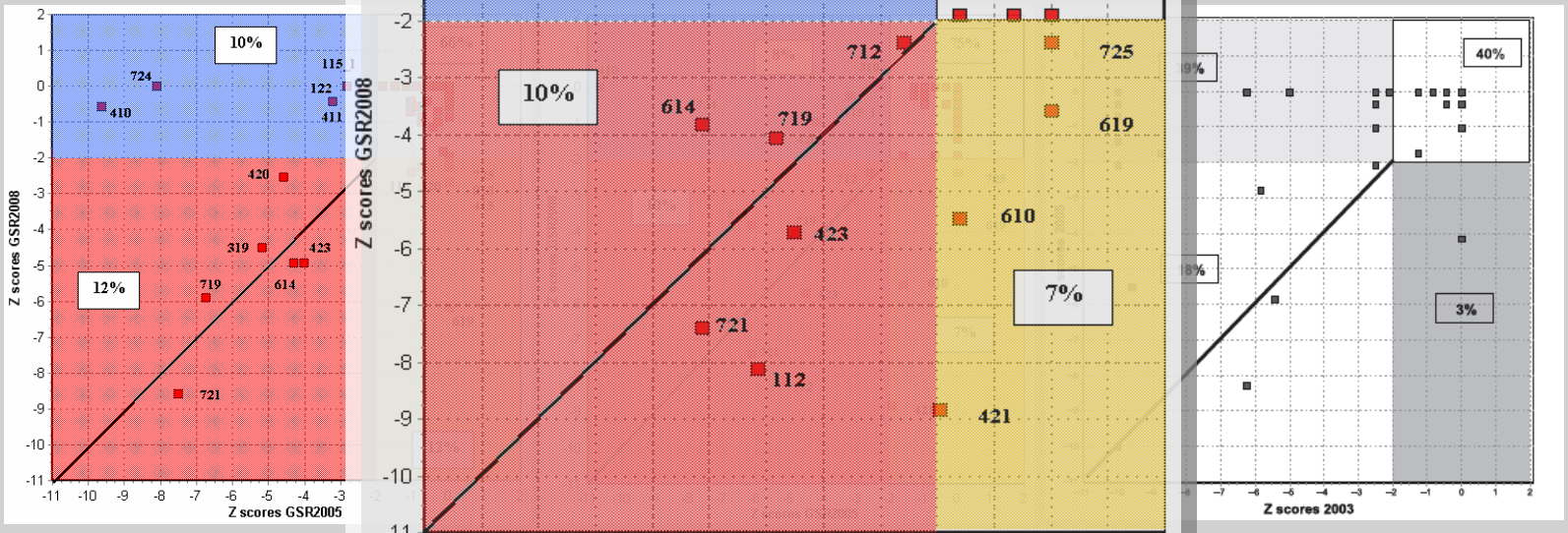


Laboratory Assessment





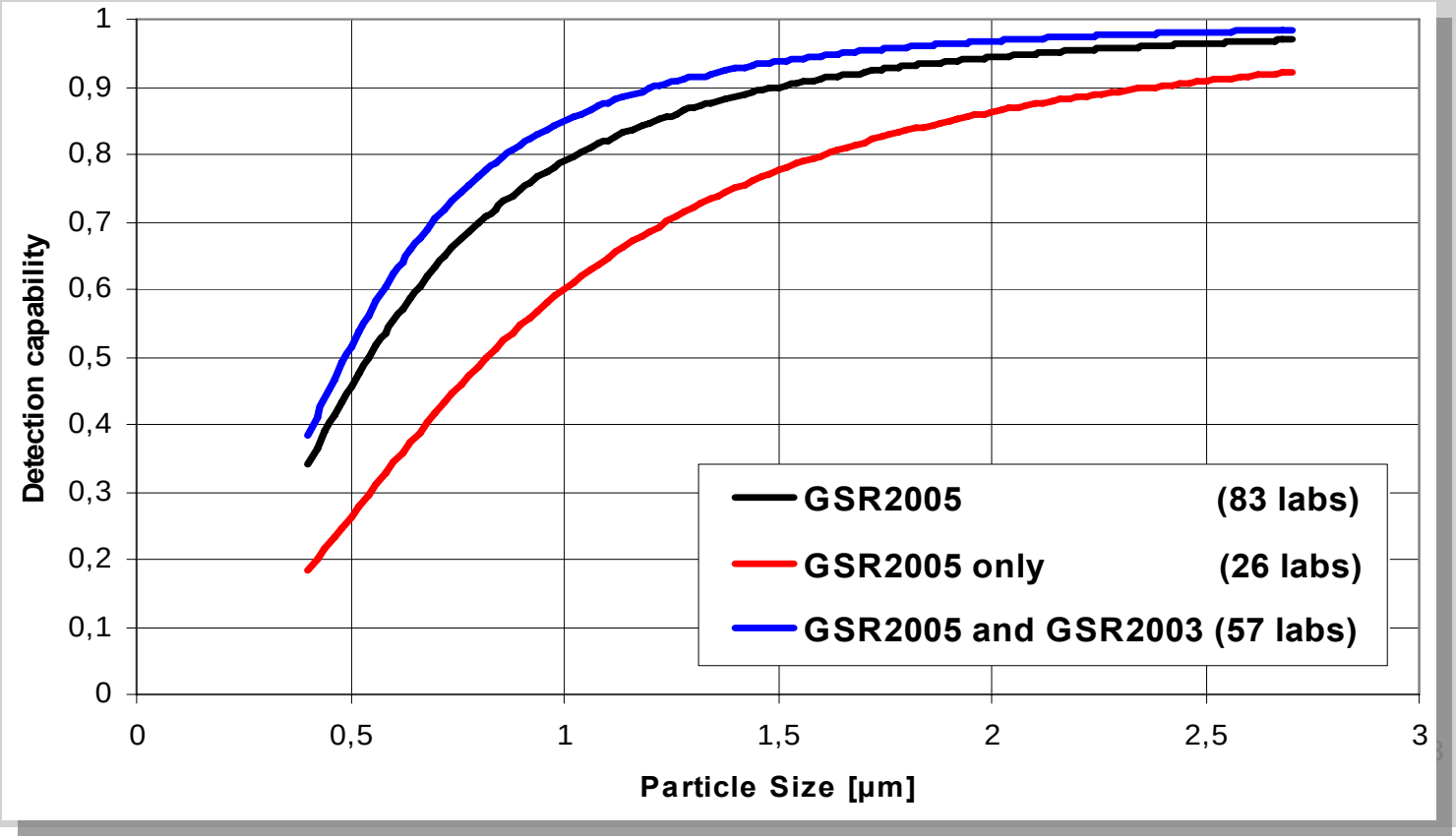
Youden-Plot



GSR2005 / GSR2008 | **GSR2008 / GSR2005** | **GSR2003 / GSR2005**

Ø ≥ 0.8 µm | **Ø ≥ 1.2 µm** | **Ø = 0.8 µm**

Method Improvement



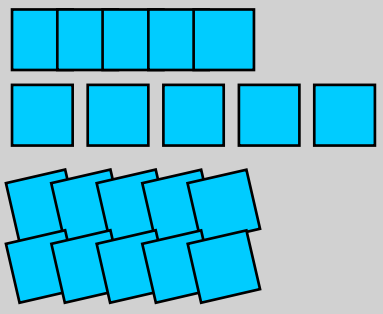
In- laboratory application

For system validation / verification

- optimisation of measurement parameters
- notification of potential systematic errors
- avoidance of unnoticed, slightly drifting parameters
- use as a standard in QA/QM
    - regular system check, documentation
    - system check after installation/upgrade/repair

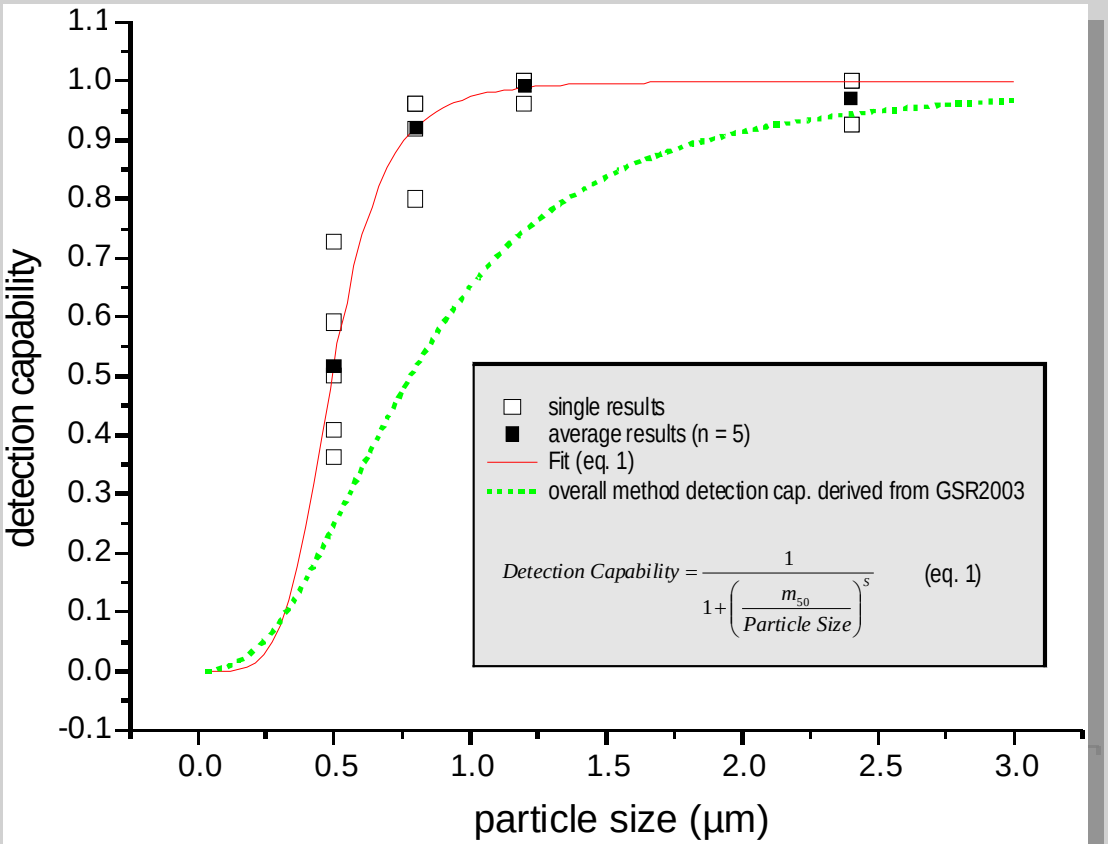
Recognition of potential errors

- incorrect stiching of fields (mechan. / electr.)
  - field overlap
  - spacing
  - WD correction



  - scan hysterethis correction (TV/point mode)
- insufficient BSE-detection/settings (brightness, contrast)
- insufficient SEM/EDX settings
  - minimum particle size, magnification, field sizes
  - EDX-calibration (GSR standard)
- instable system parameters (drift of focus, current, etc)

System Validation (III)
FEI Quanta / Oxford INCA



FEI Quanta / Oxford INCA

Outlook

GSR particle standard

- produced and distributed by PLANO GmbH

future applications

- steel industry
- MLA
- asbestos fibres

Forensische Wissenschaften

Kriminalität crime

Strafverfahren legal procedure

Kriminaltechnik

e.g. criminalistics (trace evidence), firearms and toolmarks, questioned documents, ...