Fischer Traceability Report JL-2013-10-Cd Sindelfingen, 10/09/2013

Characterization of Cd primary and secondary reference materials

Abstract:

Three new Cd primary reference standards (Cd foils) have been produced and used for the production of new Cd secondary reference standards.

Experimental details

Primary reference material (self-supporting Cd foils) has been produced on the basis of gravimetric measurements and data from the universal standard free XRF fundamental parameter method. XRF measurements have been carried out using a Fischerscope XDLM. The experimental XRF parameters are summarized in Tables I and II, for the measurement of the primary reference materials and the secondary reference standards, respectively. For the measurement of the primary standards a 25x25 matrix of 625 equidistant measurement spots covering an area of approx. 50mm x 50mm were chosen to cover the whole surface of the Cd foils. In addition the WinFTM scan mode was used to further increase the measured area. The data of each of the secondary standards to be characterized were obtained as mean value of 16 individual measurements distributed over a 4x4 matrix in a central area of 2mm x 2 mm.

Parameter	Value	Comments
Device	Fischerscope© XDLM	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	0.6 mm	
Software version	6.28 LabDB	
Spots per sample	625	Scan mode
Duration per spot	45 s	
Measured area	50 x 50 mm (BBJ, BBK, BBL)	25x25 Matrix
Anode current	300 mA	

Table I : experimental parameters for the XRF measurement of the Cu primary standards.

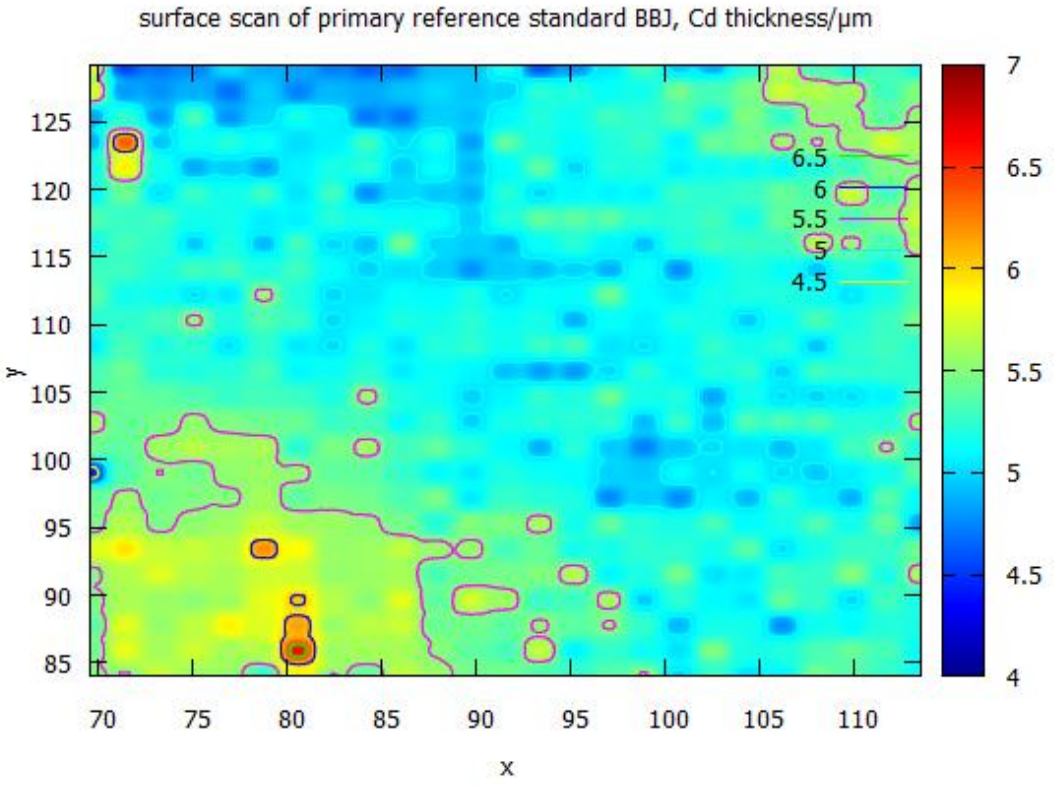
In addition weight and area of the three Cd foils for the production of primary reference standards have been measured following the standards of the DAkkS accreditation D-K-15076-01-00. The obtained mass per unit area is used as reference for the calibration of the standards free XRF values.

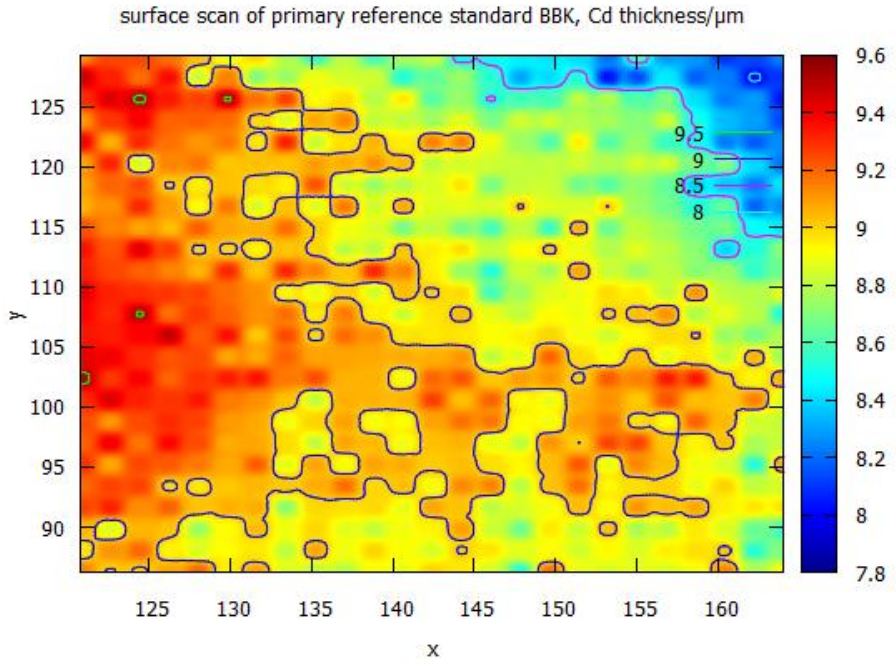
Parameter	Value	Comments
Device	Fischerscope© XDLM	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	0.6 mm	
Software version	6.28 LabDB	
Spots per sample	16	
Duration per spot	45 s	
Measured area	2 x 2 mm	4x4 Matrix
Anode current	300 mA	

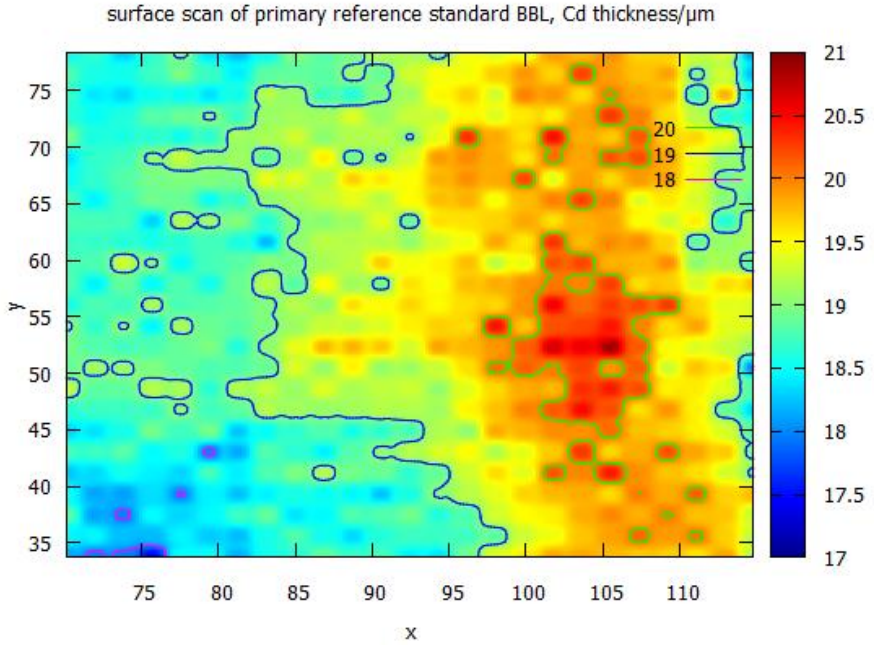
Table II : experimental parameters for the XRF measurement of the Cd secondary reference standards to be calibrated.

Data analysis and results

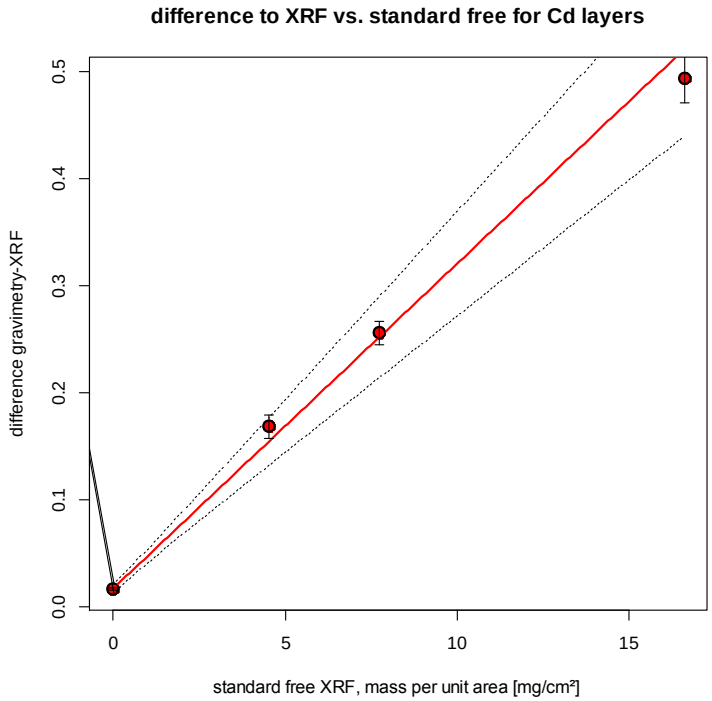
The following four figures represent heatmaps of Cd thickness values for the three primary reference standards BBJ, BBK and BBL. It is noteworthy that all samples exhibit either thickness gradients or inhomogeneous thickness distribution.




surface scan of primary reference standard BBK, Cd thickness/µm
125
120
115
110
105
100
95
90
y
125
130
135
140
145
150
155
160
x
9.5
9
8.5
8
9.6
9.4
9.2
9
8.8
8.6
8.4
8.2
8
7.8


surface scan of primary reference standard BBL, Cd thickness/µm
75
70
65
60
55
50
45
40
35
y
75
80
85
90
95
100
105
110
x
20
19
18
21
20.5
20
19.5
19
18.5
18
17.5
17

The statistical programming language R has been used to express the correlation of the difference between gravimetric and XRF measurements and the XRF measurements in terms of a linear regression.



Linear regression between the difference (gravimetry - standard free XRF) vs. standard free XRF using a linear correlation. The area between the dotted lines represents the confidence band. Fit and graphics have been produced using the statistical programming language R.

The results are summarized in table III.

Sample	Gravimetry mass per unit area mg/cm²	σ mg/cm²	XRF FP thickness µm	SE µm	mass per unit area mg/cm²	SE mg/cm²	Diff mg/cm²	σ mg/cm²
Zero	0,00	0,000	-0,02	0,001	-0,02	0,001	0,016	0,001
BBJ	4,70	0,001	5,24	0,011	4,54	0,010	0,168	0,011
BBK	7,99	0,001	8,94	0,011	7,73	0,010	0,256	0,011
BBL	17,10	0,003	19,20	0,023	16,60	0,021	0,494	0,023

Table III : Summary of experimental data from gravimetric measurements and XRF data using the standard free XRF fundamental parameter method (XRF FP) for the primary reference standards. Experimental uncertainties are given as standard deviation σ and standard error of the mean (SE). The thickness has been calculated using a density of ρ(Cd) = 8.65 g/cm³.

The correlation obtained from the regression was used to calibrate the standard free XRF values for the secondary standards. Results are summarized in Table IV.

Code	XRF FP mg/cm²	SE mg/cm²	cal mg/cm²	U(k=2) mg/cm²	cal µm	U(k=2) µm
AEAMO	4,56	0,05	4,72	0,11	5,45	0,13
AEAMP	4,53	0,04	4,68	0,09	5,41	0,10
AEAMQ	8,65	0,06	8,93	0,15	10,32	0,18
AEAMR	8,64	0,05	8,92	0,14	10,31	0,16
AEAMS	15,84	0,11	16,34	0,27	18,89	0,31
AEAMT	15,99	0,10	16,49	0,25	19,07	0,29

Table IV : Summary of experimental data from XRF measurements for the secondary Cd reference standards. Calibrated values ("cal") have been calculated using the correlation between XRF and gravimetric measurements for the primary reference standards (see Table III).

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