

Fischer Traceability Report VR 2012 07

## On the re-calibration of Ni-foil reference standards

The production of a 4 new pure Ni primary standards characterized by gravimetry /1/ is the basis of this re-calibration. These foils are checked for consistency with the previously used secondary standards<sup>1</sup>.

### Experimental

Fischerscope<sup>®</sup> XUV-S, 50 kV, 1 mm Al primary filter. Aperture  $\varnothing$ 1 mm. The size of the primary standards is 5 cm \* 5 cm. In order to get a representative mean value of the entire primary standard a scan of 8\*8 readings has been performed. The WinFTM scanning mode<sup>2</sup> has been applied to cover a large spot by a single measurement. Since the measuring distance affects the geometry factor, it must be fixed within a small tolerance. The Fischerscope<sup>®</sup> X-ray system's autofocus tool achieves an acceptable range of about 20-50  $\mu$ m.

The other standard foil samples were measured in the central area of 2 mm \* 2 mm by a map of 9 measuring points.

The measuring time of a single reading is 60 s in any case..

The foils were placed on a particular sample holder ("radiation trap") to avoid the excitation of radiation components from the surrounding chamber.

### Results of re-calibration

The 7 foil samples of the GN set 18761 and one additional single standard have been re-calibrated by the mentioned 4 new primary standards. Also the Zero (empty sample holder) has been checked.

The Tab. 1 lists both the standard free results of the primary and the secondary standards.

---

<sup>1</sup> Master standards (Gebrauchsnormale) which are traceable to gravimetric masses per unit area from former calibrations.

<sup>2</sup> The motorized table moves both in x- and y- direction with predefined velocities and predefined distances. These so-called scan parameters are defined in the WinFTM product. Here a scan area of 3 mm \* 3 mm has been used.

Tab. 1 The standard free experimental results of the foil samples under investigation are compared with the stated values. The lines 2-5 depict the data of the primary reference standard foils with the stated values from the actual mass per unit area determination acc. to the gravimetric procedure /1/. In the following lines secondary standards are displayed: nos. 6-12 GN set 18761 (from 2009) and nos. 14-18 GN set 15249 (from 2004). Line no 13 is a single standards from 2009. All the "stated values" refer to old style DKD calibrations.

	Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]
1	empty sample holder	<b>0</b>	0,0003	-	
2	<b>BAU Ni 5 070812</b>	<b>4,454</b>	4,644	-	
3	<b>BAW Ni 10 070812</b>	<b>9,026</b>	9,58	-	
4	<b>BAW Ni 15 070812</b>	<b>13,579</b>	14,34	-	
5	<b>BAX Ni 25 090812</b>	<b>22,48</b>	23,83	-	
6	ACXSU	0,973	0,972	<b>0,980</b>	0,015
7	ACXTU	1,931	1,871	<b>1,88</b>	0,019
8	ACXTB	2,507	2,488	<b>2,50</b>	0,023
9	ACXTK	4,631	4,54	<b>4,57</b>	0,033
10	ACXUE	6,539	6,60	<b>6,65</b>	0,043
11	ACXSD	18,58	17,6	<b>18,9</b>	0,104
12	ACXSN	22,01	20,7	<b>23,2</b>	0,126
13	ACLTN	5,166	5,14	<b>5,20</b>	0,036
14	AAXIQ	21,99	20,91	<b>21,2</b>	0,116
15	AAWQU	16,71	16,43	<b>16,7</b>	0,093
16	AAWPU	8,86	8,66	<b>8,78</b>	0,054
17	AAWRZ	2,54	2,49	<b>2,52</b>	0,023
18	AAXKG	1,02	1,01	<b>1,026</b>	0,015

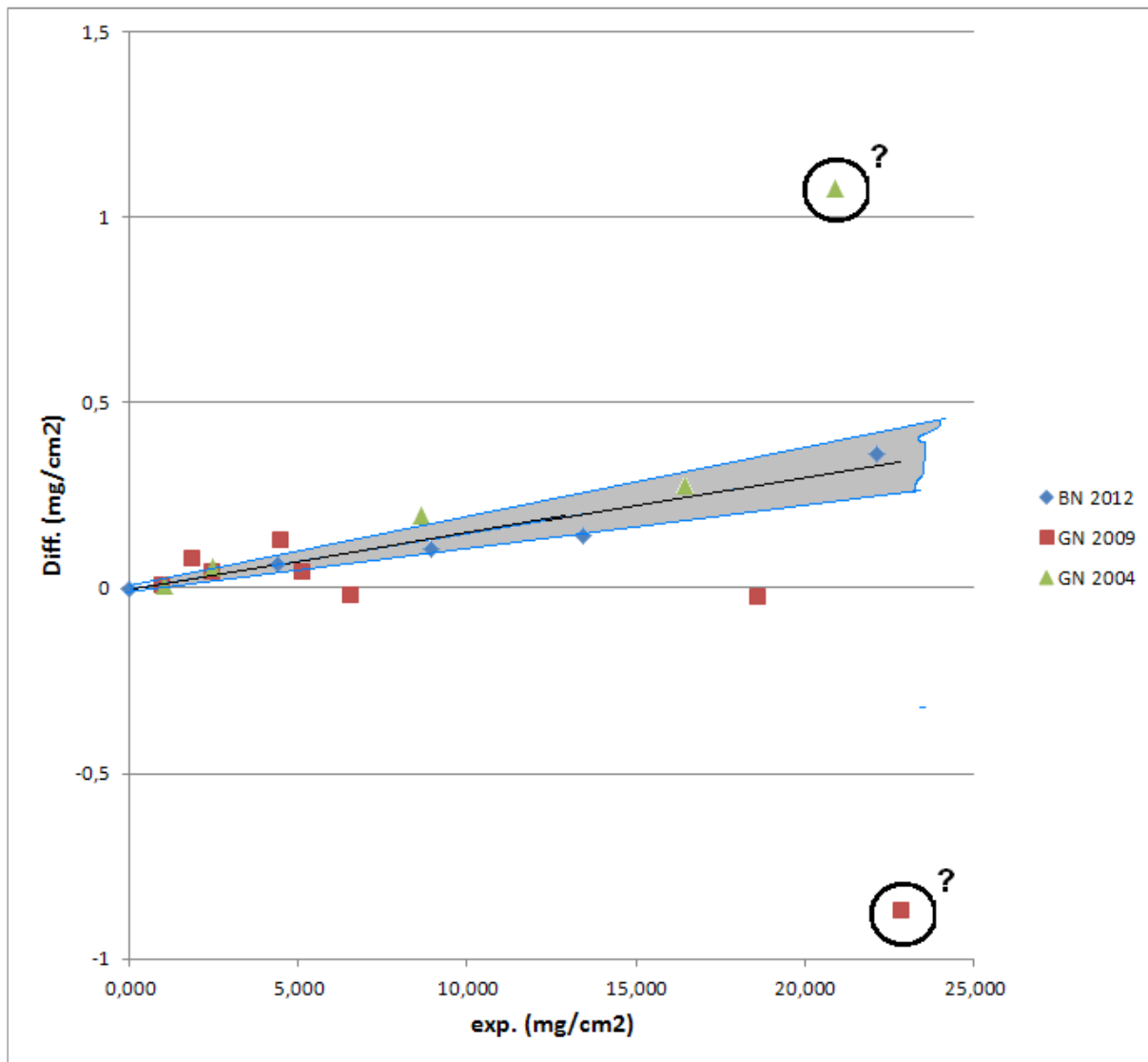


Fig. 1 Deviations of the standard free experimental readings with respect to the stated values. The blue marks denote the new primary standards and the Zero value. The red and green ones refer to the secondary master standards which are calibrated now, cf. Tab. 1. The line depicts the linear correction (1.5%) based on the Zero value and the 4 new primary standard foils.

Conclusion

The new foil standards are in good agreement with the standard free experimental values. The mean deviation is 1.5% only, cf. the blue marked dots in Fig. 1. Both GN sets are very close to the theoretical values also, except from the thickest foil (> 20 mg/cm<sup>2</sup>) in both cases. The reason of this non-consistency is not known. The calibration is quite stable and reliable due to the large number of primary standards. Also the good agreement with the old secondary foils (except from the 2 outliers) indicates a high level of confidence for the applied calibration of column 5 of Tab. 1.

The very small estimated uncertainties (column 6 of Tab. 1, about 0.5 %) are up to now the best accuracies for Fischer mass per unit area reference standards. The reasons are the quite good uniformity of the Ni foils and the relative large number of primary standards.

VR

13.08.2012

References

/1/ Reg. No. D-K-15076-01-00.

Appendix 1:

Ni/Fe

Ni/Cu/Fe

Ni/Cu

The secondary standard sets GN 18760 (Ni/Fe), GN 18762 (Ni/Cu/Fe), and GN 18759 (Ni/Cu) are re-calibrated by the same procedure as applied for the self-supporting Ni foils describe above. The measurements have been performed in the same manner.

The results are listed in Tabs. A1 and A2

Tab. A 1 *The old stated value<sup>3</sup>s and the standard free experimental results for the Ni mass per unit area of Ni/Fe samples (items 1-7 = set GN 18760 ) and Ni/Cu/Fe samples (items 8,9 = set GN 18762). The recalibration uses the same correction (+ 1.46%) of the Ni foils.*

	Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]
1	ACXSW	0,975	0,964	<b>0,978</b>	0,02
2	ACXTW	1,901	1,823	<b>1,85</b>	0,03
3	ACXTD	2,522	2,52	<b>2,56</b>	0,03
4	ACXTQ	4,561	4,5	<b>4,57</b>	0,05
5	ACXUJ	6,52	6,61	<b>6,71</b>	0,06
6	ACXSF	18,58	18,53	<b>18,8</b>	0,15
7	ACXSP	20,06	22,5	<b>22,8</b>	0,18
8	ACXSH	18,59	18,9	<b>19,2</b>	0,16
9	ACXUN	6,749	6,65	<b>6,75</b>	0,06

Tab. A2 *The old stated values and the standard free experimental results for the Ni mass per unit area of Ni/Cu samples (= set GN 18759). The recalibration uses the same correction (+ 1.46%) of the Ni foils.*

	Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]
1	ACXSV	0,956	0,94	<b>0,954</b>	0,02
2	ACXTY	1,923	1,85	<b>1,88</b>	0,03
3	ACXTC	2,524	2,5	<b>2,54</b>	0,03
4	ACXTM	4,527	4,55	<b>4,62</b>	0,05
5	ACXUF	6,545	6,57	<b>6,67</b>	0,06
6	ACXSE	18,51	18,15	<b>18,4</b>	0,15
7	ACXSO	21,97	21,94	<b>22,3</b>	0,18

VR

14.08.2012

<sup>3</sup> the "stated values" refer to old style DKD calibrations.

## Appendix 2:

Ni/Fe

Ni/Cu

The secondary standard sets GN 15721 (Ni/Fe), GN 15723 (Ni/Cu) are re-calibrated by the same procedure as applied for the self-supporting Ni foils describe above.

The measurements have been performed in the same manner.

The results are listed in Tabs. A3 and A4

Tab. A 3 *The old stated value<sup>4</sup>s and the standard free experimental results for the Ni mass per unit area of Ni/Fe samples of the GN set 15721. The recalibration uses the same correction (+ 1.46%) of the Ni foils.*

	Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]
1	ABAKH	1,070	1,056	<b>1,07</b>	0,02
2	ABADW	2,768	2,76	<b>2,80</b>	0,04
3	ACLTP	5,234	5,295	<b>5,37</b>	0,05
4	ABAEZ	8,775	8,853	<b>8,98</b>	0,08
5	ABABU	16,545	16,85	<b>17,10</b>	0,14
6	ABACT	22,562	22,91	<b>23,24</b>	0,19
7	ACLTP	5,289	5,19	<b>5,27</b>	0,05

Tab. A2 *The old stated values and the standard free experimental results for the Ni mass per unit area of the GN set 15723. The recalibration uses the same correction (+ 1.46%) of the Ni foils.*

	Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]
1	ACLTR	5,291	5,325	<b>5,40</b>	0,05
2	ABAKF	1,039	1,02	<b>1,03</b>	0,02
3	ABADU	2,701	2,68	<b>2,72</b>	0,04
4	ACLTS	5,18	5,09	<b>5,16</b>	0,05
5	ABAEX	8,988	8,987	<b>9,12</b>	0,08
6	ABABS	16,56	16,61	<b>16,85</b>	0,14
7	ABACR	22,47	22,24	<b>22,56</b>	0,18

VR

15.08.2012

<sup>4</sup> the "stated values" refer to old style DKD calibrations.

Appendix 3:  
Ni/Fe

The secondary standard set GN 15719 (Ni/Fe) is re-calibrated by the same procedure as applied for the self-supporting Ni foils describe above.

The measurements have been performed in the same manner.

The results are listed in Tab. A5

*Tab. A 5 The old stated value<sup>5</sup>s and the standard free experimental results for the Ni mass per unit area of Ni/Fe samples of the GN set 15721. The recalibration uses the same correction (+ 1.46%) of the Ni foils. The mass per unit area of the last two columns are converted into thickness values using the density  $\rho=8.9 \text{ g/cm}^3$ .*

Sample / Code	stated value [mg/cm <sup>2</sup> ]	exp. [mg/cm <sup>2</sup> ]	re-calibrated [mg/cm <sup>2</sup> ]	measuring uncertainty (k=1) [mg/cm <sup>2</sup> ]	re-calibrated [μm]	measuring uncertainty (k=1) [μm]
ABAKJ	1,05	1,04	<b>1,055</b>	0,02	46,7	1,0
ABADY	2,68	2,68	<b>2,72</b>	0,04	120,1	1,6
ABAFB	9,14	9,06	<b>9,19</b>	0,08	406,7	3,7
ABABN	16,73	16,67	<b>16,91</b>	0,14	748	6,2
ABACV	22,52	22,38	<b>22,71</b>	0,18	1005	8,1

VR  
15.08.2012

---

<sup>5</sup> the "stated values" refer to old style DKD calibrations.