Fischer Traceability Report SD 2012 02

Recertification of Au on Pd on Ni master standard foils

The Helmut Fischer GmbH applies master reference standards to quantify “standard calibration set” products. This report describes the recertification of the master reference standards for Au/Pd/Ni foils.

The mass per unit areas of the foils are traceable to reference materials with low uncertainties:

- Au values: traceable to Au/Pd/Ni/PCB Master reference materials (this means tracable to a combination of gravimetry /1/, FP-based XRF analysis /2/, and Rutherford backscattering /3/. Cf. Fischer Traceability Report VR 2011 01.

- Pd values: traceable to Au/Pd/Ni/PCB Master reference materials (this means a combination of gravimetry /1/, FP-based XRF analysis /2/, and Rutherford backscattering /3/) and self-supporting Pd foils quantified according to a gravimetric procedure accredited by either DKD or DAkkS /1/. Cf. Fischer Traceability Report SD 2012 01.

- Ni values traceable to self-supporting Ni foils (Sets 18761and 15722) quantified according to a gravimetric procedure accredited by either DKD or DAkkS /1/.

Experimental

Fischerscope® X-RAY XDAL®, 50 kV, 1 mm Al primary filter. aperture Ø 0.6 mm, DefMA Au/Pd/Ni/ (with background correction), Pd was measured, evaluation without Pd L radiation. The self-supporting foils were measured with a special “radiation trap” sample holder.
All samples were measured with 9 measurements with 120 s uniformly distributed over an area of 2 mm x 2 mm.
Since the measuring distance affects the geometry factor, it must be fixed within a small tolerance. The Fischerscope X-Ray system’s autofocus tool achieves an acceptable constant measuring distnce of about ±20 µm.
The measurements were repeated several times to ensure consistency. For control purposes, various uncoated samples were checked.

Results of recalibration

All values converted from the mass per unit area data with the density for Au = 19,3 g/cm^3 , Pd = 12 g/cm^3 and Ni = 8,9 g/cm^3 The given uncertainties are standard measuring uncertainties for a confidence level of 68,3 % (k =1).

Code	Au in nm			Pd in nm			Ni in nm		
	specified	New value	u (k = 1)	specified	New value	u (k =1)	specified	New value	u (k =1)
ADBFR	29	29	1	-	-	-	4380	4402	80
ADDZM	-	-	-	99	97	3,5	4275	4336	80
ADBFA	60	61	2	21	21	1,5	4224	4293	80
ADBGK	100	101	2	-	-	-	4287	4345	80
ADDZV	48	48	2	250	244	5	4506	4514	80
ADBES	63	62	2	62	62	3	4503	4488	80
ADDZU	47	48	2	100	98	3,5	4303	4343	80
ADBFH	-	-	-	21	21	1,5	4280	4331	80
ADDZL	-	-	-	247	244	5	4429	4417	80
ADBFM	-	-	-	61	62	3	4320	4344	80
Ni foil ABYBP	-	-	-	-	-	-	3900	3921	80

Discussion

As shown in the table above, in most cases the recertified values are very close to the old values. The differences are usually in the same order of magnitude as the measuring uncertainties. Although the reasons for these discrepancies cannot be explained in each case, the spatial inhomogeneity is probably an important issue which has not previously been accounted for. Consequently, the recalibrated results are mean values valid for the very restricted area of 2 mm x 2 mm.

Further basic reference material will be produced in future to achieve improved data with smaller measuring uncertainties.

References

/1/ Reg. No. D-K-15076-01-00.
/2/ V. Rößiger and B. Nensel, in “Handbook of practical X-Ray fluorescence analysis”, Springer 2006, p. 554.
/3/ S. Dill and V. Rößiger, Circuit world, 37 (2011), 2, 20.