

# Re-evaluation of Cr/Fe reference standards

## Abstract:

Cr/Fe reference standards have been re-evaluated using new traceable reference standards (see Traceability Report TraceabilityReport\_JL2012\_11\_CrCu\_CrFe).

## Experimental details

The availability of new Cr/Fe reference standards which are traceable to ICP-OES measurements (see Fischer Traceability Report JL-2012-11-Cr/Cu-Cr/Fe) permits a re-evaluation of existing Cr/Fe secondary reference standards and unambiguous traceability in accordance with the DAkkS accreditation D-K-15076-01-00. The experimental parameters are summarized in Table I. For the measurement of the traceable Cr/Fe reference standards and the standards to be re-evaluated a 4x4 matrix of 16 equidistant measurement spots covering an area of 2 mm x 2 mm in the central region of the primary standards was defined.

Parameter	Value	Comments
Device	Fischerscope <sup>®</sup> XDV-SDD	
Voltage, Filter	50keV, Ni 10 µm primary filter	
Aperture collimator	0.2 mm	
Software version	6.29	
Spots per sample	16	
Duration per spot	60 s	
Measured area	2 x 2 mm	4x4 Matrix
Anode current	1000	

Table I : experimental parameters for the XRF measurement of the traceable Cr/Fe reference standards and standards to be re-measured.

In order to increase the sensitivity for Cr thicknesses of > 15µm the measuring distance was set to 13.6 mm.

## Results

The mean value of the 16 individual measurements using the universal standard free XRF fundamental parameter method for each of the reference standards and their nominal values from Fischer

Traceability Report JL-2012-11-Cr/Cu-Cr/Fe were used for calibration applying the internal calibration routine in the WinFTM software. Nominal and measured values are summarized in Table II.

**Calibration**

Sample	Code	nominal [mg/cm <sup>2</sup> ]	measured [mg/cm <sup>2</sup> ]
2a10	ADYCB	1.48	1.483
4a10	ADYCL	2.89	2.883
8a10	ADYCV	6.03	6.01
12a10	ADYDF	8.99	9.31
16a10	ADYDP	12.16	12.93
20a10	ADYDZ	9.91	10.38
24a10	ADYEJ	17.18	19.18

Table II : Mean XRF values of the 16 individual measurements per standard obtained from the universal standard free XRF fundamental parameter method compared to their nominal values from Fischer Traceability Report JL-2012-11-Cr/Cu-Cr/Fe.

The obtained calibration was used to re-evaluate the reference standards listed in Table III. The overall agreement is excellent except for the standard with code ABSKX

Sample	Soll [μm]	XRF / Cr [μm]	U(k=2) [μm]	XRF / Cr mg/cm <sup>2</sup>	σ mg/cm <sup>2</sup>	#	σ <sub>mean</sub> mg/cm <sup>2</sup>	rho Cr g/cm <sup>3</sup>
Fe	0	-0,005	0,001	-0,003	0,001	16	0,0003	7,19
M22	0,346	0,350	0,002	0,251	0,003	16	0,0007	7,19
M28	0,615	0,611	0,003	0,439	0,004	16	0,0010	7,19
M35	2,07	2,105	0,006	1,514	0,008	16	0,0019	7,19
M42	4,346	4,339	0,017	3,120	0,023	16	0,0056	7,19
M52	7,748	7,620	0,033	5,479	0,044	16	0,0111	7,19
M55	8,877	8,789	0,026	6,319	0,035	16	0,0088	7,19
M59	11,55	11,63	0,056	8,364	0,076	16	0,0190	7,19
M64	14,55	14,86	0,158	10,682	0,214	16	0,0536	7,19
M72	16,59	17,03	0,214	12,247	0,291	16	0,0727	7,19
Fe	0	0,00	0,001	0,000	0,001	16	0,0003	7,19
Cr	∞	29,08	0,610	20,905	0,828	16	0,2069	7,19
AAQEX	0,61	0,61	0,004	0,439	0,005	16	0,0014	7,19
AAQEX	1,92	1,96	0,007	1,412	0,010	16	0,0025	7,19
AAQEX	7,7	7,72	0,015	5,551	0,020	16	0,0049	7,19
ABSKX	16,9	20,91	0,290	15,035	0,393	16	0,0983	7,19

Table III : Mean XRF values of the 16 individual measurements per standard obtained from the universal standard free XRF fundamental parameter method compared to their nominal values from Fischer Traceability Report JL-2012-11-Cr/Cu-Cr/Fe.

Sindelfingen, 11/30/2012

Dr. Jörg Leske