

Certified Reference Material (CRM)
Certificate of Chemical Analysis

CRM AlZn-G3
(Zinc 99.7)

Laboratory means (4 values), mass content in %

No.	Al	Fe	Pb
1	0.1446	-----	0.00183
2	0.1451	-----	0.00200
3	0.1488	0.0410	0.00202
4	0.1489	0.0411	0.00213
5	0.1492	0.0416	0.00213
6	0.1506	0.0417	0.00220
7	0.1514	0.0417	0.00224
8	0.1525	0.0418	0.00235
9	0.1556	0.0418	0.00240
10	0.1607	0.0418	
<i>M</i>	0.1507	0.0416	0.00214
<i>s_M</i>	0.0048	0.0004	0.00018
\bar{s}_i	0.0023	0.0003	0.00014

M : Mean of the intralaboratory means

s_M : Standard deviation of the intralaboratory means

\bar{s}_i : averaged repeatability standard deviation (square root of the mean
of laboratory variances)

The laboratory mean values have been examined statistically to eliminate outlying values. Where a "-----" appears in the table it indicates that an outlying value has been omitted by either the Cochran or Grubbs test.

CERTIFIED VALUES (mass fraction in %)

	Al	Fe	Pb
M(M)	0.151	0.0416	0.00214
U	0.004	0.0003	0.00012

U is the estimated expanded uncertainty with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, comprising contributions from the certification interlaboratory comparison and inhomogeneity contributions.

Berlin, November 2019

The certificate is valid until 31.12.2040.

Description of the sample

The sample is available in the form of discs (38 mm diameter and 30 mm height).

Sputter factors and density

CRM **AlZn-G3** is part of a series of 11 CRMs intended for calibration of GDOES spectrometers. For validation of the quantitative depth profiling methods, it is recommended to use for density of these CRMs the values from the following table. The sputter factors and densities listed below are non-certified (only informative) values.

CRM	AlZn-G1	AlZn-G2	AlZn-G3	AlZn-G4	AlZn-G5	AlZn-G6
Sputter factor	7.21	6.25	6.08	5.77	4.97	4.48
Density in g/cm ³	7.13	7.12	7.12	7.11	7.01	6.70

CRM	AlZn-G7	AlZn-G8	AlZn-G9	AlZn-G10	AlZn-G11
Sputter factor	3.80	3.52	1.97	0.81	0.72
Density in g/cm ³	6.19	6.59	5.36	3.87	3.70

The sputter factors were determined at IFW Dresden as described in L. Wilken et al., J. Anal. At. Spectrom., 2003, 18, 1141-1145 by 10 independent crater volume measurements after 600 s sputtering at 700 V and 20 mA using a 4 mm DC source. The sputtering factors are defined by the ratio of the sputtering rate of the sample to pure iron using the same discharge voltage and current. For pure iron, the sputter rate of NIST 1761 was measured after 900 s sputtering at 700 V and 20 mA and a sputter factor of 0.941 was defined for this sample in order to agree with the until now used sputter factor for this sample. The density of NIST 1761 is 7.8004 ± 0.0028 g/cm³.

In addition, the sputter factors were determined gravimetrically by an inter-laboratory experiment (participating laboratories see below). The results of this experiment agreed well with the volumetric results of IFW Dresden. The densities of the samples were measured at Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin.

Description of the sample

This reference material was prepared in accordance with the recommendations set out in ISO Guides 30 – 35 and issued by the German Iron and Steel CRM Working Group.

The German Iron and Steel CRM Working Group is composed of Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin Max-Planck-Institut für Eisenforschung GmbH (MPI), Düsseldorf Steel institute VDEh, Düsseldorf

Sale of the reference material: Bundesanstalt für Materialforschung und -prüfung (BAM), Richard-Willstätter-Straße 11, 12489 Berlin (www.webshop.bam.de).

Intended use & stability

The CRM is intended for establishing or checking the calibration of spark and glow discharge optical emission and X-ray spectrometers for the analysis of samples of similar matrix composition. It is also intended for the determination of the sputter rate in glow discharge spectrometry. The minimum sample size for wet chemical analysis is 0.2 g.

Before use, the surface of the material must be prepared by milling or turning on a lathe. For wet chemical analysis chips have to be prepared by turning or milling of the sample surface.

It will remain stable, provided that the material is stored in a dry and clean environment at room temperature. Transport has to be done under normal ambient conditions.

Traceability

The traceability of CRM AlZn-G3 has been established in accordance with principles of ISO Guides 30 – 35 and the International vocabulary of basic and general terms in metrology.

The characterisation of this material has been achieved by inter-laboratory study, each laboratory using the method of their choice, details of which are given above. These methods are either stoichiometric analytical techniques or methods which are calibrated against primary substances.

Participating laboratories

AMCO united, Duisburg (Germany)
 ArcelorMittal Eisenhüttenstadt, Forschungs- und Qualitätszentrum GmbH, Eisenhüttenstadt (Germany)
 BMW AG, München (Germany)
 Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)
 Boliden Odda AS, Odda (Norway)
 Boliden Kokkola AS, Kokkola (Finland)
 Hydro Aluminium Rolled Products GmbH, R&D-Bonn (Germany)
 Leibniz-Institut für Festkörper- und Werkstoffforschung (IFW) Dresden (Germany)
 thyssenkrupp Steel Europe AG, Duisburg (Germany)
 Weser-Metall GmbH, Nordenham (Germany)
 voestalpine Stahl GmbH, Linz (Austria)

Participating laboratories (sputter factors)

ArcelorMittal Eisenhüttenstadt, Forschungs- und Qualitätszentrum GmbH, Eisenhüttenstadt (Germany)
 Fyzikální ústav AV ČR, v. v. i., Prague (Czech Republic)
 Leco Corporation, St. Joseph, MI (USA)
 Leibniz-Institut für Festkörper- und Werkstoffforschung (IFW) Dresden (Germany)
 Spectruma Analytik GmbH, Hof (Germany)
 thyssenkrupp Steel Europe AG, Duisburg (Germany)
 voestalpine Stahl GmbH, Linz (Austria)
 Volkswagen AG Wolfsburg (Germany)

Methods used for chemical analysis

Element	Line number	Method/Detection
Al	1, 2, 3, 4, 5, 4, 7, 8, 10 9	ICP-OES FAAS
Fe	3, 4, 5, 6, 8, 9, 10 7	ICP-OES Spectrophotometry
Pb	1, 4, 5, 6, 8 2, 3, 9 7	ICP-OES ICP-MS ETAAS

Abbreviations: ETAAS – Electrothermal atomic absorption spectrometry
 FAAS – Flame atomic absorption spectrometry
 ICP-OES – Inductively coupled plasma - optical emission spectrometry
 ICP-MS – Inductively coupled plasma - mass spectrometry

Further information

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by the German CRM working group may be obtained from the address above.

The German Iron and Steel CRM Working Group

The Working Group is composed of
 Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin
 Max-Planck-Institut für Eisenforschung GmbH (MPI), Düsseldorf
 Steel institute VDEh, Düsseldorf

Dr. Sebastian Recknagel
 Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin