National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 628

Spectrographic Zinc-Base Die-Casting Alloy D

This Standard Reference Material (SRM) is intended primarily for evaluating chemical and instrumental methods of analysis of zinc-base die-casting alloys. SRM 628 is one of a series of reference materials (SRMs 625 through 630) for this purpose. A unit of SRM 628 consists of a bar segment approximately 44 mm square and 19 mm thick. The metallurgical condition is that resulting from a continuous chill casting process.

Certified Values: The certified values for 11 elements are listed in Table 1. The test methods used for certification are listed in Table 2. All values are reported as mass fractions [1] calculated as the unweighted mean of the mean values from the individual laboratories. The uncertainty listed with each value is an expanded uncertainty (approximately 95 % confidence level [2]) the standard deviation of the mean of means and calculated in accordance with the method in ISO and NIST Guides [3].

Table 1. Certified Values with Expanded Uncertainties

Element	Mass Fraction (%)	Element	Mass Fraction (%)
Aluminum	4.59 ± 0.06	Magnesium	0.0094 ± 0.0015
Cadmium	0.0040 ± 0.0011	Manganese	0.0091 ± 0.0009
Chromium	0.0087 ± 0.0009	Nickel	0.030 ± 0.002
Copper	0.611 ± 0.017	Silicon	0.008 ± 0.002
Iron	0.066 ± 0.002	Tin	0.0017 ± 0.0002
Lead	0.0045 ± 0.0004		

Expiration of Certification: The certification of this SRM is valid indefinitely provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is damaged or otherwise altered. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by R.E. Michaelis of the National Bureau of Standards (NBS) Spectrographic Standards Laboratory and R.K. Bell of the NBS Nonferrous Laboratory.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Date: 20 September 2005
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

The certified portion of each sample is that part included in a region between 5 mm and 17 mm from each side of the square sample. The center core (5 mm square) and the outer portion (from the edge inward 5 mm) may differ in composition and should not be used. Within the bounds given above, the entire thickness (19 mm) of the sample is certified. Each packaged sample has been prepared by finishing the test surface using a milling machine. The user must determine the optimum surface preparation procedure for each analytical technique. The user is cautioned to use care when either resurfacing the block or performing additional polishing as these processes may contaminate the surface. For optical emission spectrometric methods, it is recommended that a single determination be based on the average value from at least six (6) individual "burns" taken within the certified portion of the sample. Specimens prepared for chemical methods of test should consist of either a cross-section piece or large chips taken from the certified portion of the sample. Particular care should be given to obtain complete dissolution of the specimen because an appreciable part of some elements in the material are not readily soluble in simple acid mixtures.

Table 2. Analytical Methods

Element	Methods
Aluminum	Hg Cathode – 8-Hydroxyquinoline Gravimetric Method 8-Hydroxyquinoline Photometric Method Al_2O_3 Method
Cadmium	Polarigraphic Method Sulfide Gravimetric Method Spectrographic Method
Chromium	Diphenylcarbazide Photometric Method
Copper	Polarigraphic Method HBr Photometric Method Neocuprine Photometric Method H_2S Separation and Electrodeposition Electrolytic Method
Iron	Ortho-Phenanthroline Photometric Method NH_4CNS Photometric Method Iron reduced with H_2S, Zn, or lead amalgam and titrated with $KMnO_4$
Lead	Polarigraphic Method Spectrographic Method Electrolytic Method Dithizone Photometric Method
Magnesium	Diammonium Phosphate Method
Manganese	KIO_4 Photometric Method
Nickel	Dimethylglyoxime Photometric Method
Silicon	$HClO_4$ Dehydration Molybdisilicic Acid Photometric Method H_2SO_4 Dehydration Molybdenum Blue Photometric Method
Tin	Tin reduced with Ni and titrated with KIO_3 Distillation – Dithiol-butyl acetate – Photometric Method Dithiol Photometric Method Tin coprecipitated with MnO_4, reduced with Pb and titrated with iodine

Material Preparation: The material for the preparation of this SRM was melted and cast at the National Lead Co., (Chicago, IL) under a cooperative program between NBS and General Motors Corporation. Homogeneity testing of selected samples from the SRM lot was performed by R.C. Frank, J.E. Dallemand, and D.L. Fry of the Research Laboratories Division of General Motors Corporation and by the NBS Spectrographic Standards Laboratory.

User Experience with SRM 628: This alloy is known to be heterogeneous on a microscopic level. The heterogeneity is observable when the alloy is measured using spark source optical emission spectrometry (SS-OES), for example. The behavior of the material was demonstrated by one of the cooperating laboratories in order to define the certified portion of a single unit of the SRM. To indicate the level of heterogeneity, Table 3 lists the relative standard deviation of at least six individual spark burns for the certified elements. The information provided in Table 3 is for guidance and is not certified.

Table 3. Repeatability of Measurements from Individual Spark Burns from a Single Unit of SRM 628

Element	Relative Standard Deviation (%) ($n \geq 6$)
Al	2.8
Cd	4.1
Cr	17.0
Cu	3.3
Fe	19.0
Pb	3.8
Mg	13.5
Mn	21.0
Ni	19.0
Si	86.0
Sn	4.7

Cooperating Laboratories

Cooperative analyses for certification were performed in the following laboratories:

General Motors Corp., Chemistry Department; Detroit, MI (USA); M.D. Cooper, R.L. Chance, A.H. Jones, R.E. Kohn, and R.B. Loranger

The New Jersey Zinc Co.; Palmerton, PA (USA); S.N. Roeder

Apex Smelting Co.; Cleveland, Ohio (USA); R.L. Vitek and J.W. Mierzwa

Hudson Bay Mining and Smelting Co. Ltd.; Flin Fon, Manitoba (Canada); D.J. Robertson, D.J. Sample, and L.S. Creighton

Metal & Thermit Corp., Research Laboratory; Rahway, NJ (USA); M. Farnsworth and J.S. Pekola

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; U.S. Government Printing Office: NIST Special Publication 811 (1995).

[2] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

[3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Certificate Revision History: 20 September 2005 (This technical revision adds instructions for use of the material and editorial revisions to reflect program and organizational changes at NIST); 05 June 1996 (Editorial revision to reflect program and organizational changes at NIST); 24 April 1964 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.