



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 630

#### Zinc-Base Die-Casting Alloy F (block form)

This Standard Reference Material (SRM) is intended primarily for evaluating chemical and instrumental methods of analysis of zinc-base die-casting alloys. SRM 630 is one of a series of reference materials (SRMs 630 through 630) for this purpose. A unit of SRM 630 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 Mass Fraction Values:** The measurand is the total elemental content for each constituent in SRM 630. 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 the ISO/JCGM Guide [3]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the “true” value based on the results of analyses performed at NIST and collaborating laboratories. Metrological traceability is to the derived SI unit for mass fraction (expressed as a percent).

**Expiration of Certification:** The certification of **SRM 630** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Use”). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

The original characterization of SRM 630 was performed in 1964. Overall direction and coordination of the technical measurements leading to certification of this SRM were performed by R.E. Michaelis of NIST [formerly the National Bureau of Standards (NBS)] Spectrographic Standards Laboratory and R.K. Bell of the NBS Nonferrous Laboratory.

Analyses for certification were performed by the following collaborating 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, L.S. Creighton; and Metal & Thermit Corp., Research Laboratory (Rahway, NJ, USA), M. Farnsworth and J.S. Pekola.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Gaithersburg, MD 20899  
Certificate Date: 19 October 2016  
*Certificate Revision History on Last Page*

Steven J. Choquette, Director  
Office of Reference Materials

## 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.

**Material Preparation<sup>(1)</sup>:** 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. Dallemund, and D.L. Fry of the Research Laboratories Division of General Motors Corporation and by the NBS Spectrographic Standards Laboratory.

Table 1. Certified Mass Fraction Values with Expanded Uncertainties for SRM 630

Element	Mass Fraction (%)	Element	Mass Fraction (%)
Aluminum (Al)	4.30 ± 0.04	Magnesium (Mg)	0.030 ± 0.002
Cadmium (Cd)	0.0048 ± 0.0006	Manganese (Mn)	0.0106 ± 0.0008
Chromium (Cr)	0.0031 ± 0.0004	Nickel (Ni)	0.0027 ± 0.0003
Copper (Cu)	0.976 ± 0.016	Silicon (Si)	0.022 ± 0.003
Iron (Fe)	0.023 ± 0.001	Tin (Sn)	0.0040 ± 0.0003
Lead (Pb)	0.0083 ± 0.0007		

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<sup>(1)</sup> Certain commercial equipment, instruments, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 2. Analytical Methods

Element	Methods
Aluminum	Hg Cathode – 8-Hydroxyquinoline Gravimetric Method
	8-Hydroxyquinoline Photometric Method
	Al <sub>2</sub> O <sub>3</sub> Method
Cadmium	Polarigraphic Method
	Sulfide Gravimetric Method
	Spectrographic Method
Chromium	Diphenylcarbazide Photometric Method
Copper	Polarigraphic Method
	HBr Photometric Method
	Neocuprine Photometric Method
	H <sub>2</sub> S Separation and Electrodeposition
	Electrolytic Method
Iron	Ortho-Phenanthroline Photometric Method
	NH <sub>4</sub> CNS Photometric Method
	Iron reduced with H <sub>2</sub> S, Zn, or lead amalgam and titrated with KMnO <sub>4</sub>
Lead	Polarigraphic Method
	Spectrographic Method
	Electrolytic Method
	Dithizone Photometric Method
Magnesium	Diammonium Phosphate Method
Manganese	KIO <sub>4</sub> Photometric Method
Nickel	Dimethylglyoxime Photometric Method
Silicon	HClO <sub>4</sub> Dehydration
	Molybdisilicic Acid Photometric Method
	H <sub>2</sub> SO <sub>4</sub> Dehydration
	Molybdenum Blue Photometric Method
Tin	Tin reduced with Ni and titrated with KIO <sub>3</sub>
	Distillation – Dithiol-butyl acetate – Photometric Method
	Dithiol Photometric Method
	Tin coprecipitated with MnO <sub>4</sub> , reduced with Pb and titrated with iodine

**User Experience with SRM 630:** 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. Informational values cannot be used to establish metrological traceability.

Table 3. Information Values for Repeatability of Measurements from Individual Spark Burns from a Single Unit of SRM 630

Element	Relative Standard Deviation (%) (n ≥ 6)
Al	4.2
Cd	4.2
Cr	57.0
Cu	10.0
Fe	53.0
Pb	8.0
Mg	6.6
Mn	34.0
Si	18.0
Sn	11.0

## REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at <http://www.nist.gov/pml/pubs/sp811/index.cfm> (accessed Oct 2016).
- [2] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).
- [3] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at [http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Oct 2016); 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://www.nist.gov/pml/pubs/index.cfm> (accessed Oct 2016).

**Certificate Revision History:** 19 October 2016 (Title change; editorial changes) 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 of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*