NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 1229
Zirconium (Sn-Fe-Cr) Alloy UNS R60804
(block form)
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM®) are intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of zirconium and its alloys. They can be used to validate value assignment of a laboratory's in-house reference materials.

Description: A unit of SRM 1229 consists of one block of approximate dimensions of 31 mm square by 18.5 mm thick, produced by electron beam cutting from plate stock.

Certified Values: A NIST certified value is the present best estimate of the true value [1]. The certified values for SRM 1229 are shown in the table below and are metrologically traceable to the International System of Units (SI) derived unit of mass fraction, expressed as milligrams per kilogram.

Certified Values for Elements in SRM 1229

Element	Mass Fraction[(a)] (mg/kg)	Element	Mass Fraction[(a)] (mg/kg)
Chromium (Cr)	1539 ± 23	Nickel (Ni)	17.8 ± 1.7
Cobalt (Co)	5.12 ± 0.41	Niobium (Nb)	99.9 ± 8.3
Hafnium (Hf)	31.7 ± 2.8	Tin (Sn)[(b)]	19900 ± 550
Iron (Fe)	1601 ± 54	Tungsten (W)	22.1 ± 3.9
Lead (Pb)	8.0 ± 1.1		

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value [2–4]. The true value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. For guidance in propagating this uncertainty, see reference 5.

[(b)] The overlined digit is the least significant digit of the certified value for Sn.

Non-Certified Values: Non-certified values for elements in SRM 1229 are provided in Appendix A.

Additional Information: Values of potential interest to users are provided in Appendix B.

Period of Validity: The certification of **SRM 1229** is valid indefinitely, within the measurement uncertainty specified. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified. Periodic recertification of this SRM is not required.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Office of Reference Materials

Storage: Store the SRM in its original container in a dry location (relative humidity <50 %) at room temperature (25 °C ± 10 °C).

Use: The test surface is the side opposite to the labeled surface, which includes the SRM number. The certified and non-certified values are valid for the entire thickness of the unit. However, the user is cautioned not to measure disks less than 2 mm thick when using X-ray fluorescence spectrometry. The test method used to characterize material homogeneity indicates sampling a minimum recommended mass of 25 mg for a single specimen or, for direct measurement techniques, the minimum recommended measurement area is a total of 13 mm^2 covered by one or more independent measurements. Each packaged block has been prepared by finishing the test surface using a milling machine. The other surfaces were created by electron beam cutting of the block from the original plate material. The user must determine the correct surface preparation procedure for each analytical technique. The user is cautioned to use care when either resurfacing the disk or performing additional polishing as these processes may contaminate the surface. It was found by NIST that abrasive paper must be changed frequently during surface grinding. Used paper loses its ability to remove contaminants from the surface of the alloy.

Source: The material for SRM 1229 was obtained from a commercial manufacturer of zirconium and its alloys. The material was cut and packaged at NIST.

Analysis: Measurements used to certify SRM 1229 were performed at NIST and a collaborating laboratory. Test methods included combustion, inductively coupled plasma mass spectrometry, inductively coupled plasma optical emission spectrometry, inert gas fusion, neutron activation analysis, titrimetry, and X-ray Fluorescence spectrometry.

Notice to Users: NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and a laboratory's in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sharpless, K.E.; Sieber, J.R.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136 2021 Edition; National Institute of Standards and Technology, Gaithersburg, MD (2021) available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jan 2024).

[2] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall: London, UK (1993).

[3] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley: Hoboken, NJ (1992).

[4] DerSimonian, R.; Laird, N.; *Meta-analysis in Clinical Trials*; Controlled Clinical Trials, Vol. 7, No. 3, pp. 177-188 (1986).

[5] Possolo, A.M.; *Evaluating, Expressing, and Propagating Measurement Uncertainty for NIST Reference Materials*; NIST Special Publication (NIST SP) 260-202; National Institute of Standards and Technology, Gaithersburg, MD (2020) available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-202.pdf (accessed Jan 2024).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, Maryland 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are best estimates based on currently available information. However, they do not meet NIST's criteria for certification [1]. Non-certified values cannot be used to establish metrological traceability to the SI or another higher-order reference system. These mass fraction values are metrologically traceable to the values for calibration standards used by the collaborating laboratory for one or more test methods. They may be used to establish traceability of test results to these values for this reference material. Non-certified values could be used in situations where metrological traceability to the SI is not necessary, for example method harmonization, method development or quality control.

Non-certified values for SRM 1229 are provided below.

Non-Certified Values for Elements in SRM 1229

Element	Mass Fraction[(a)] (mg/kg)
Aluminum (Al)	20 ± 5
Boron (B)	0.6 ± 0.1
Copper (Cu)	5.31± 0.27
Manganese (Mn)	7.2 ± 1.2
Phosphorus (P)	87.2 ± 8.3
Tantalum (Ta)	174 ± 21
Titanium (Ti)	12.6 ± 1.9
Vanadium (V)	21 ± 2

[(a)] These values are expressed as $x \pm 2u(x)$, where x is the value and $u(x)$ is the standard uncertainty of x. The standard uncertainty represents the combined effects of measurement repeatability and calibration uncertainty for a single test method and material composition variance from multiple independent specimens. While the best estimates of the values lie within the intervals $x \pm 2u(x)$, these intervals may not include the true values. For guidance on using and propagating this uncertainty, see reference 5.

Period of Validity: The non-certified values are valid indefinitely within the measurement uncertainties specified. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Values of Potential Interest to Users: Approximate mass fraction values for additional elements in SRM 1229 were obtained by one test method. The values have not been confirmed nor have the uncertainties of the values been assessed. This information is provided to help the user assess possible measurement interferences. This information cannot be used to establish metrological traceability to the SI or another higher-order reference system. Do not use these values to calibrate or to validate a test method.

Element	Mass Fraction[(a)] (mg/kg)	Element	Mass Fraction[(a)] (mg/kg)
Antimony (Sb)	<10	Palladium (Pd)	20
Bismuth (Bi)	<1	Ruthenium (Ru)	<50
Cadmium (Cd)	0.07	Samarium (Sm)	<5
Calcium (Ca)	<10	Silicon (Si)	36
Chlorine (Cl)	<1	Silver (Ag)	<1
Gadolinium (Gd)	<2.5	Sodium (Na)	<5
Hydrogen (H)	14	Thallium (Tl)	<25
Magnesium (Mg)	<10	Thorium (Th)	<3.5
Molybdenum (Mo)	9	Uranium (U)	<1
Nitrogen (N)	21	Yttrium (Y)	<5
Oxygen (O)	940	Zinc (Zn)	<50

[(a)] The mass fraction values given as 'less than' (<) values represent estimates of the limits of quantification for the test methods used to attempt to determine the amounts present in SRM 1229.

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