Analytical Reference Materials International

Certificate of Analysis Certified Reference Material

Grade: **17-4 PH / UNS S17400**Part Number (Q.A. NO.): **IARM 23D**

Interpretation of Data

- 1. Certified values listed below reflect analysis results submitted by qualified analytical laboratories using a combination of methods and instrumentation that emulate actual methods and instrumental techniques currently utilized in the analytical community and are reported as % wt. unless otherwise noted.
- 2. Any data reported and enclosed by a parentheses () is a "best estimate" and is NOT CERTIFIED. This data could not be quantified sufficiently for certification. It was however, reported by enough laboratories to be considered as potentially present in the matrix of the material being examined.
- 3. The "Inter-laboratory Analysis Program" (ILAP) utilized in the establishment of the data are an ongoing program with permanent membership. Certain elements may be selected by a consensus of the members for more extensive testing. Therefore the data in **brackets [] indicates further testing is in process.**
- 4. The "±Estimated Uncertainty" is enclosed by a parentheses () below the individual element's concentration and is based on a Confidence Interval at 95%. Included in this estimated uncertainty, are the combined effects of method imprecision, material inhomogeneity, and any bias between methods.

Important: A "User Registration Card" accompanies all shipments. This card should be completed immediately upon receipt of materials with the appropriate user information. This is the only way in which ARMI can guarantee customer updates or possible data modifications!

| Aluminum 0.005 (0.001) | Antimony | <u>Arsenic</u> (0.005) | Boron 0.0019 (0.0001) | Calcium 0.0013 (0.0002) | Carbon 0.038 (0.001) | <u>Cerium</u> | Chromium 15.16 (0.02) | Cobalt 0.041 (0.001) | <u>Copper</u> 3.27 (0.01) |
|------------------------------|-----------------------------|-------------------------------------|-------------------------------------|----------------------------------|-------------------------------|------------------------------|------------------------------|------------------------------|---------------------------------|
| <u>Hydrogen</u> | <u>Lead</u> (0.001) | <u>Magnesium</u> (0.0004) | Manganese 0.52 (0.005) | Molybdenum 0.159 (0.001) | Nickel 4.88 (0.02) | Niobium 0.29 (0.004) | Nitrogen 0.037 (0.001) | Oxygen 0.0033 (0.0002) | Phosphorus 0.022 (0.001) |
| <u>Selenium</u> (0.001) | Silicon 0.490 (0.003) | <u>Sulfur</u> 0.0006 (0.0002) | <u>Tantalum</u> 0.002 (0.001) | <u>Tin</u> 0.0059 (0.0002) | <u>Titanium</u> 0.003 (0.001) | Tungsten 0.011 (0.002) | Vanadium 0.053 (0.001) | <u>Zinc</u> (0.001) | Zirconium (0.001) |

The laboratories participating in the "Inter-Laboratory Analysis Program" (ILAP) and certification of this material are as follows:

Alcoa Howmet, Dover Alloy - Dover, NJ ATI Specialty Materials, Lockport - Lockport, NY

Crucible Industries - Syracuse, NY

Laboratorio Prove Materiali S. Marco srl - Schio, Italy

Latrobe Specialty Metals - Latrobe, PA PM Kalco, Inc - Wheatland, PA Anderson Laboratories, Inc. - Greendale, WI ATI Specialty Materials, Monroe - Monroe, NC

Ellwood National Steel - Irvine, PA Laboratory Testing, Inc. - Hatfield, PA NSL Analytical Services - Cleveland, OH Timken Steel Corp. - Canton, OH

Traceability: All members of the "Inter-Laboratory Analysis Program" (ILAP) listed above validate test methods and instrument performance utilizing SRMs produced by the National Institute of Standards and Technology, (NIST) as well as other CRMs and RMs produced by recognized Certifying Bodies from around the world. The specific SRMs, CRMs, and RMs applicable to the material covered by this certificate are: NIST 1261A, 1767, BCS 466/1, 468, BS 11, 60C, 61C, 185A, 8620B, IARM 23B, BS 88G, 174PHB, CSN-4, IN ALV 9903B, LECO 502-257, 502-455, NIST 363, 898, IARM 23B, ALPHA AR660, AR869, LECO 501-644, 501-992, 502-702, NIST 345A, 361, 3101A, 3103A, 3107, 3109A, 3128, 3131A, 3149, 3155, 3161A, 3162A, 3163, 3168A, 3169, MBH 13XPH1M, 13XPH17700A, PH25143A, ALPHA AR511, AR654, AR669, AR881, LECO 501-502, IARM 23C, BS 88E, ALPHA AR872, LECO 502-257, NIST 16F, 1249, 1262, C2400, BAS 346A, IARM 23B, LECO 501-644, IARM 23C, ALPHA AR654, AR661A, AR881, AR891, NIST 16F, 362, 368, C1151, 1152, C1153, C1154, 1155, 1160, 1161, 1162, 1163, 1164, 1185, 1187, 1219, 1260, 1261, 1261A, 1262, 1262B, 1263, 1264, C1287, C1288, C1289, 1754, 1760, 1761, 1762, 1763, 1764, 1765, 1766, 1767, 2166, C2400, BAS 61, 401/1, 401/2, 402/1, 403/1, 404/1, 405/1, 406/1, 407/2, 408/1, 409/1, 410/2, 451, 452, 453, 454, 456, 457, 458, 459, 460, 465/1, 466/1, 470, 472, 473, 474, 475, JSS ST-01, 168-4, 169-4, 170-4, 171-4, 190-1, 191-1, 192-1, 193-1, 194-1, 195-1, 650-1102, NIST 339, 345, IARM 2C, 4B, 11C, 15B, 21B, 23B, 23C, LECO 501-644, 501-679, 502-195, 502-449, IARM 23C, BS 88C, 88E, 88F, 88G, 185, 185A, 192, 9622, NIST 867, 3101A, 3107, 3109A, 3128, 3131A, 3161A, BS 17-4PH B, AL RV495, RV496, RV499, LECO 501-643, IARM 22B, 327A, ASM 0621, 0622, 0821, 0822, CARTECH 077, ALPHA AR669.

A specific line of traceability is established to NIST and other Certifying Bodies for those elements that are noted as "Certified Values" on the Certificates of Analyses referenced above.

See Reverse Side for Statistical Data and Additional Information Regarding this Material.

The following data and accompanying statements represent all pertinent information reported in the ILAP as it applies to the chemical characterization of this material as of 10/10/2014.

| | | _ | _ | | _ | _ | | | | | | _ | _ | _ | |
|-----------|--------|--------|--------|--------|---------|--------|--------|--------|--------|--------|--------|--------|--------|---------|----------|
| 23D | Al | В | С | Co | Cr | Cu | Mn | Мо | N | Nb | Ni | 0 | P | S | Se |
| 1 | 0.0045 | 0.0017 | 0.037 | 0.040 | 15.22 | 3.28 | 0.510 | 0.160 | 0.036 | 0.290 | 4.860 | 0.0034 | 0.023 | 0.0010 | 0.0003 |
| 2 | 0.0057 | 0.0020 | 0.0374 | 0.0395 | 15.211 | 3.232 | 0.522 | 0.158 | 0.0368 | 0.3036 | 4.889 | 0.0037 | 0.0224 | 0.0001 | 0.000003 |
| 3 | 0.0038 | 0.0018 | 0.037 | 0.0411 | 15.158 | 3.292 | 0.524 | 0.161 | 0.0377 | 0.2986 | 4.838 | 0.0031 | 0.0221 | 0.0005 | 0.001 |
| 4 | 0.0048 | 0.0021 | 0.0407 | 0.039 | 15.193 | 3.272 | 0.5319 | 0.1565 | 0.0379 | 0.301 | 4.893 | 0.0037 | 0.021 | 0.0002 | 0.0005 |
| 5 | 0.0059 | 0.0019 | 0.0376 | 0.0429 | 15.156 | 3.248 | 0.5183 | 0.1575 | 0.0381 | 0.3070 | 4.883 | 0.0030 | 0.0202 | 0.0007 | 0.0010 |
| 6 | 0.0048 | 0.0021 | 0.040 | 0.0413 | 15.179 | 3.280 | 0.522 | 0.158 | 0.0365 | 0.281 | 4.85 | 0.0031 | 0.0245 | 0.0007 | |
| 7 | 0.0042 | 0.0018 | 0.0405 | 0.0398 | 15.094 | 3.269 | 0.5307 | 0.1598 | 0.0381 | 0.2936 | 4.936 | 0.0033 | 0.0216 | 0.00118 | |
| 8 | 0.0033 | 0.0017 | 0.0406 | 0.0397 | 15.120 | 3.250 | 0.524 | 0.159 | 0.0351 | 0.296 | 4.896 | 0.0033 | 0.021 | 0.00047 | |
| 9 | 0.0033 | 0.0022 | 0.0373 | 0.0434 | 15.125 | 3.246 | 0.531 | 0.160 | 0.0376 | 0.2856 | 4.940 | | 0.0228 | 0.0006 | |
| 10 | 0.0063 | | 0.0352 | 0.0406 | 15.177 | 3.305 | 0.5188 | 0.1567 | 0.0396 | 0.2945 | 4.917 | | 0.0209 | 0.00067 | |
| 11 | 0.0050 | | 0.0374 | 0.0409 | 15.103 | 3.249 | 0.5380 | 0.159 | 0.0391 | 0.2834 | 4.832 | | 0.0218 | 0.00022 | |
| 12 | 0.0054 | | 0.0357 | 0.040 | 15.210 | 3.262 | 0.520 | 0.158 | 0.0378 | 0.295 | 4.88 | | 0.023 | 0.001 | |
| 13 | | | | 0.0423 | 15.21 | 3.31 | 0.527 | 0.1573 | 0.035 | 0.299 | 4.880 | | 0.0202 | 0.0007 | |
| 14 | | | | | 15.149 | 3.279 | 0.5062 | | 0.0382 | 0.2996 | | | | | |
| | | | | | | | | | | | | | | | |
| Mean | 0.0047 | 0.0019 | 0.0380 | 0.0408 | 15.1646 | 3.2695 | 0.5231 | 0.1585 | 0.0374 | 0.2949 | 4.8841 | 0.0033 | 0.0219 | 0.0006 | 0.0006 |
| STDV. | 0.0010 | 0.0002 | 0.0019 | 0.0014 | 0.0422 | 0.0233 | 0.0085 | 0.0014 | 0.0014 | 0.0076 | 0.0339 | 0.0003 | 0.0013 | 0.0003 | 0.0004 |
| Certified | 0.005 | 0.0019 | 0.038 | 0.041 | 15.16 | 3.27 | 0.52 | 0.159 | 0.037 | 0.29 | 4.88 | 0.0033 | 0.022 | 0.0006 | (0.001) |
| 95% C.I. | 0.001 | 0.0001 | 0.001 | 0.001 | 0.02 | 0.01 | 0.005 | 0.001 | 0.001 | 0.004 | 0.02 | 0.0002 | 0.001 | 0.0002 | |
| Methods | X,I,O | I,O | C,O | X,I,O | X,I,O | X,I,O | X,I,O | X,I,O | F,O | X,I,O | X,I,O | F | X,I,O | C,G,O | X,G,I,O |

Legend: W = Classical, C = Combustion, F = Fusion, A = AA or GFAA, I = ICP or DCP, D = DC Arc, O = AES, X = XRF, G = GDAES or GDMS, H = Hollow Cathode AES

| 23D | Si | Sn | Та | Ti | ٧ | W | Ag | As | Bi | Ca | H | Mg | Pb | Zn | Zr |
|-----------|--------|---------|--------|---------|--------|--------|----------|---------|----------|---------|--------|----------|---------|---------|---------|
| 1 | 0.491 | 0.0064 | 0.0010 | 0.0029 | 0.0540 | 0.0114 | < 0.0001 | 0.0060 | <0.0010 | 0.0012 | 0.0001 | 0.0004 | 0.0018 | 0.0008 | 0.0010 |
| 2 | 0.4835 | 0.0057 | 0.0015 | 0.0024 | 0.0525 | 0.0146 | 0.0001 | 0.0042 | <0.00002 | 0.0011 | | 0.0005 | 0.0005 | 0.0007 | 0.00004 |
| 3 | 0.4913 | 0.0057 | 0.0010 | 0.0017 | 0.0529 | 0.0084 | | 0.0038 | 0.000001 | 0.00147 | | 0.0004 | 0.0006 | 0.0005 | 0.0001 |
| 4 | 0.491 | 0.0061 | 0.0011 | 0.0034 | 0.054 | 0.0081 | | 0.0050 | | 0.0011 | | 0.0002 | 0.0005 | 0.0007 | 0.0004 |
| 5 | 0.4877 | 0.0057 | 0.0024 | 0.0026 | 0.0537 | 0.0122 | | 0.0034 | | 0.00102 | | | 0.0022 | | 0.0019 |
| 6 | 0.491 | 0.0053 | 0.0021 | 0.0019 | 0.051 | 0.0069 | | | | 0.0017 | | | 0.0028 | | 0.0012 |
| 7 | 0.489 | 0.0060 | | 0.0036 | 0.0522 | 0.011 | | | | 0.0013 | | | 0.00051 | | 0.0013 |
| 8 | 0.4930 | 0.0058 | | 0.0028 | 0.0544 | 0.0091 | | | | | | | 0.0007 | | 0.001 |
| 9 | 0.4906 | 0.0061 | | 0.0034 | 0.0535 | 0.0149 | | | | | | | | | |
| 10 | 0.498 | 0.0062 | | 0.0032 | 0.052 | 0.011 | | | | | | | | | |
| 11 | 0.4837 | 0.0055 | | 0.0044 | 0.054 | 0.011 | | | | | | | | | |
| 12 | | 0.006 | | | 0.0517 | 0.0120 | | | | | | | | | |
| 13 | | 0.0061 | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Mean | 0.4900 | 0.0059 | 0.0015 | 0.0029 | 0.0530 | 0.0115 | 0.0001 | 0.0045 | 0.0000 | 0.0013 | 0.0001 | 0.0004 | 0.0012 | 0.0007 | 0.0009 |
| STDV. | 0.0041 | 0.0003 | 0.0006 | 0.0008 | 0.0011 | 0.0031 | | 0.0010 | | 0.0002 | | 0.0001 | 0.0009 | 0.0001 | 0.0006 |
| Certified | 0.490 | 0.0059 | 0.002 | 0.003 | 0.053 | 0.011 | | (0.005) | | 0.0013 | | (0.0004) | (0.001) | (0.001) | (0.001) |
| 95% C.I. | 0.003 | 0.0002 | 0.001 | 0.001 | 0.001 | 0.002 | | | | 0.0002 | | | | | |
| Methods | X,I,O | X,G,I,O | X,I,O | X,G,I,O | X,I,O | X,I,O | | X,G,I,O | | G,I,O | | G,I | G,I,O | G,I | X,G,I,O |

Legend: W = Classical, C = Combustion, F = Fusion, A = AA or GFAA, I = ICP or DCP, D = DC Arc, O = AES, X = XRF, G = GDAES or GDMS, H = Hollow Cathode AES

The International Standards Organization (ISO) definitions, expressed in ISO Guide 30-1992 list the following:

<u>Certifying Body:</u> Any technically competent body (organization or firm, public or private) that issues a reference material certificate, which provides the information, detailed in ISO Guide 31. The only generally accepted certifying body in the United States for primary standards - Standard Reference Materials (SRM) is the U. S. Department of Commerce, National Institute of Standards & Technology, (NIST), Gaithersburg, MD. All other certifying bodies in the United States produce Reference Materials (RM) or Certified Reference Materials (CRM).

Reference Material (RM): Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

<u>Certified Reference Material (CRM):</u> Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure, which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

Inter-Laboratory Analysis Program (ILAP): Although ASTM Standard E691-87 applies to inter-laboratory studies to "Determine the Precision of a Single Test Method", it is also a well thought out and logical plan for conducting an inter-laboratory program involving multiple techniques. Therefore, the planning, conducting, analyzing, protocol, and treatment of data resulting from this inter-laboratory program were performed utilizing the guidelines established in ASTM E691-87.

Methods of Analysis: In view of the fact, that the "Inter-laboratory Analysis Program" entails a wide variety of materials, no single analytical method would provide optimum data results. Therefore, the methods utilized were a combination of ASTM Standard Methods for classical wet chemistry, ICP, AA, Optical Emission, and X-Ray spectrometric methods. The determinations for Carbon, Sulfur, Nitrogen, and Oxygen are the result of combustion and OE instrument procedures.

Expiration of Certification: The certification of this IARM is valid indefinitely, within the uncertainty specified, provided the IARM is handled and stored in accordance with the instructions stated on this certificate. The certification is nullified if the IARM is damaged, contaminated, otherwise modified, or used in a manner for which it was not intended.

Instructions for Use: The test surface is the side opposite to the labeled surface, which includes the IARM number. The entire thickness of the unit is certified. However, the user is cautioned not to measure disks less than 2 mm thick when using X-ray fluorescence spectrometry. Each packaged disk has been prepared by finishing the test surface using a lathe. 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. When not in use, the material should be stored in a cool, dry location. This material was tested using both the solid disks and chips prepared from the disks. The certified values are considered representative of the overall average composition of the material. Chips are not intended to be used for Oxygen analysis.

Selection of Materials: A "batch" or "series" is defined as a single bar of one continuous length and heat. The majority of materials are in wrought condition; other methods of manufacture are utilized as a less desirable resort. ILAP samples are taken by removing a section, a minimum of, every one-twelfth of total length from the entire bar. A portion of the section is converted to chips and thin (pin) disk for analysis by classical wet chemistry, ICP, AA, and combustion procedures, and the balance remains as a thick disk for OES and X-Ray analysis. This systematic sampling procedure results in the homogeneity being reflected as a product of the overall statistics and certified data. This method of homogeneity testing is in accordance with ISO Guide 34, regarding the systematic selection and testing of a representative number of units for the assessment of homogeneity.

Daniel Geist, Operations Manager Analytical Reference Materials International, Part of LGC Standards

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