Analytical Reference Materials International

Certificate of Analysis Certified Reference Material



Grade: 15-5 PH

Part Number (Q.A. NO.): IARM 22B

Certificate Date: 06/18/1998

Certificate No.: 22B-06181998-IARM-F

Revision Date: 07/10/2007

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!

| <u>Aluminum</u> 0.004 (0.0010) | <u>Boron</u> (0.0007) | <u>Carbon</u> 0.045 (0.001) | <u>Cobalt</u> 0.08 (0.001) | <u>Chromium</u> 14.29 (0.02) | <u>Copper</u> 3.25 (0.03) | <u>Manganese</u> 0.53 (0.00) |
|--------------------------------------|-------------------------------------|------------------------------------|--------------------------------------|-------------------------------------|-------------------------------------|------------------------------------|
| <u>Molybdenum</u> 0.35 (0.003) | <u>Nitrogen</u> 0.012 (0.001) | <u>Niobium</u> 0.301 (0.003) | <u>Nickel</u> 4.81 (0.03) | <u>Oxygen</u> 0.001 (0.000) | Phosphorus 0.022 (0.001) | <u>Sulfur</u> 0.001 (0.000) |
| <u>Silicon</u> 0.41 (0.01) | <u>Tantalum</u> (0.010) | <u>Tin</u> 0.010 (0.001) | <u>Titanium</u> 0.003 (0.0009) | <u>Vanadium</u> 0.054 (0.002) | <u>Tungsten</u> 0.028 (0.002) | <u>Zirconium</u> |

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

| AK Steel - Middletown, OH | Allegheny Ludlum Corp Brackenridge, PA |
|--|---|
| Allvac Steel - Lockport, NY | Anderson Laboratories, Inc Greendale, WI |
| Armco, Inc Butler, PA | Bodycote Metals Analysis, Inc Huntington Park, CA |
| Chicago Spectro Service Laboratories - Chicago, IL | Jorgensen Forge Corp Seattle, WA |
| Laboratory Testing, Inc Dublin, PA | Lockheed Martin Astronautics - Littleton, CO |
| Talley Metals Technology, Inc. | Timken Latrobe Steel - Latrobe, PA |
| Wisconsin Centrifugal, Inc Waukesha, WI | |

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 1226, 1261A, 1262A, 1265A, C1151, C1152, C1153, C1154, 1155, 1193, 1194, 1195, 1230, C2400, JSS 650, 651, 652, 653, 654, 655, BCS331, 332, 333, 334, 335, 336, 337, 338, ARMCO 8709, 8710, 8711, 8712, MBH14933, 14934, 14935, BSC 401/1, 402/1, 403/1, 404/1, 405/1, 406/1, 407/1, 408/1, 409/1, 410/1, IARM 1B, 2B, 4B, 5B, 9B, 11B, 152A, 157A162A, 163A, 205A, 27B, 28B, 29B, 30B, 31B, 32B, 33B, 34B, 35B, 36B, 48B, 49B, 155A, 156A, 164A, 165A, 166A, 167A, 168A, 169A, 170A, 171A, 172A, BS185, BS17-4PHA, BS17-4PHB, NIST 348A, 121D, 133B, 345, 101E, 2159, LECO 501-644, 501-643, TSNH-500, TASN-100, TBA-250, TBIN-250, TCAN-500, TMGN-500, TPBN-250, TSEN-100, TTEN-100, TZRN-250, NIST 1763,1765,1766,368,1154,LECO 501-551, NIST 15L, 343A, 73C, IARM 22A, BS185A, PH3G, LECO 501-550, IARM 22A, BS 185, 17-4A, 17-4B, RV 9902A, 9902B, 9902C, LECO 501-674, 501

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 07/10/2007.

| 22B | AI | В | С | Co | Cr | Cu | Mn | Мо | Ν | Nb | Ni | 0 | Р | S | Si |
|---|--------|----------|---------|--------|---------|--------|--------|---------|---------|--------|--------|--------|--------|----------|--------|
| 1 | 0.005 | 0.0007 | 0.044 | 0.08 | 14.255 | 3.197 | 0.552 | 0.351 | 0.011 | 0.3 | 4.88 | 0.0012 | 0.019 | 0.0008 | 0.414 |
| 2 | 0.0042 | 0.00059 | 0.046 | 0.0835 | 14.25 | 3.17 | 0.5342 | 0.3433 | 0.0132 | 0.2974 | 4.768 | 0.001 | 0.022 | 0.0011 | 0.4211 |
| 3 | 0.0048 | 0.0005 | 0.046 | 0.08 | 14.30 | 3.29 | 0.5199 | 0.3515 | 0.0128 | 0.3039 | 4.752 | 0.001 | 0.0202 | 0.001 | 0.42 |
| 4 | 0.0055 | 0.00126 | 0.043 | 0.081 | 14.297 | 3.32 | 0.541 | 0.349 | 0.01227 | 0.297 | 4.87 | 0.0008 | 0.021 | 0.0014 | 0.4 |
| 5 | 0.004 | 0.0003 | 0.0463 | 0.0819 | 14.31 | 3.268 | 0.532 | 0.346 | 0.01095 | 0.299 | 4.83 | 0.0012 | 0.0216 | 0.0014 | 0.412 |
| 6 | 0.0028 | 0.001 | 0.0483 | 0.081 | 14.314 | 3.214 | 0.533 | 0.351 | 0.0127 | 0.302 | 4.804 | 0.0015 | 0.0226 | 0.0008 | 0.423 |
| 7 | | | 0.04475 | 0.082 | 14.265 | 3.243 | 0.532 | 0.349 | 0.01167 | 0.305 | 4.73 | 0.0010 | 0.023 | 0.001 | 0.411 |
| 8 | | | 0.043 | 0.08 | 14.32 | 3.240 | 0.536 | 0.353 | 0.012 | 0.302 | 4.822 | 0.0003 | 0.021 | 0.001 | 0.41 |
| 9 | | | 0.0443 | 0.081 | 14.27 | 3.27 | 0.528 | 0.347 | 0.0123 | 0.297 | 4.82 | 0.0007 | 0.022 | 0.0014 | 0.409 |
| 10 | | | 0.042 | 0.08 | | 3.243 | 0.535 | 0.343 | 0.0136 | 0.3075 | 4.88 | | 0.0222 | 0.0015 | 0.4 |
| 11 | | | 0.0459 | | | 3.215 | 0.536 | 0.3403 | 0.0127 | | 4.773 | | 0.024 | 0.00128 | |
| 12 | | | 0.0454 | | | 3.341 | 0.533 | | | | 4.78 | | 0.023 | 0.001 | |
| 13 | | | 0.0463 | | | | | | | | 4.757 | | 0.022 | 0.0011 | |
| 14 | | | | | | | | | | | 4.93 | | 0.019 | | |
| | | | | | | | | | | | | | | | |
| Mean | 0.0044 | 0.0007 | 0.0450 | 0.0812 | 14.2871 | 3.2509 | 0.5343 | 0.3476 | 0.0123 | 0.3011 | 4.8140 | 0.0010 | 0.0216 | 0.0012 | 0.4120 |
| STDV. | 0.0009 | | 0.0017 | 0.0012 | 0.0271 | 0.0499 | 0.0075 | 0.0041 | 0.0008 | 0.0036 | 0.0587 | 0.0003 | 0.0014 | 0.0002 | 0.0080 |
| Certified | 0.004 | (0.0007) | 0.045 | 0.08 | 14.29 | 3.25 | 0.53 | 0.35 | 0.012 | 0.301 | 4.81 | 0.001 | 0.022 | 0.001 | 0.41 |
| 95% C.I. | 0.0010 | | 0.001 | 0.001 | 0.02 | 0.03 | 0.00 | 0.003 | 0.001 | 0.003 | 0.03 | 0.000 | 0.001 | 0.000 | 0.01 |
| Methods | X,I,O | O,I,D | С | X,I,O | X,W,O | X,I,O | X,I,O | X,I,O | F | X,I,O | X,W,O | F | X,I,O | X,C | X,W,O |
| Methods: W = Classical, C = Combustion, F = Fusion, A = AA or GFAA, I = ICP or DCP, D = DC Arc, O = OE, X = XRF, G=GDMS | | | | | | | | | | | | | | | |
| 22B | Sn | Ti | V | W | Fe | As | Bi | Ca | Н | Mg | Pb | Se | Та | Te | Zr |
| | 0.044 | 0.001 | 0.0500 | 0.0070 | | 0.0000 | 0.0000 | 0.00.40 | | 0.0005 | 0.0005 | 0.0005 | 0.000 | 0.000107 | |

| 220 | 0.11 | | | | | 7.0 | | ou | | mg | | 00 | 14 | | |
|-----------|---|---------|--------|--------|---------|----------|----------|--------|--|--------|----------|-----------|---------|----------|---------|
| 1 | 0.011 | 0.004 | 0.0503 | 0.0279 | 75.777 | 0.0026 | 0.0009 | 0.0042 | | 0.0035 | 0.0025 | < 0.0005 | 0.002 | 0.000127 | <0.01 |
| 2 | 0.009 | 0.005 | 0.0558 | 0.0253 | | 0.0022 | < 0.001 | 0.0002 | | <0.001 | <0.001 | 0.0069 | 0.0132 | | 0.0008 |
| 3 | 0.0104 | 0.0016 | 0.052 | 0.025 | | <0.01 | 0.000182 | | | 0.0002 | < 0.0001 | <0.000027 | 0.010 | | 0.0016 |
| 4 | 0.0095 | 0.0032 | 0.0542 | 0.0310 | | 0.005349 | | | | | 0.0006 | | 0.0102 | | <0.01 |
| 5 | 0.009 | 0.002 | 0.054 | 0.031 | | | | | | | < 0.0001 | | 0.010 | | 0.0009 |
| 6 | 0.0107 | 0.00468 | 0.0563 | 0.031 | | | | | | | 0.000487 | | 0.0145 | | |
| 7 | 0.01 | 0.004 | 0.054 | 0.03 | | | | | | | | | | | |
| 8 | 0.01 | 0.002 | 0.057 | 0.0263 | | | | | | | | | | | |
| 9 | | 0.003 | 0.056 | | | | | | | | | | | | |
| 10 | | | 0.051 | | | | | | | | | | | | |
| 11 | | | 0.0556 | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| Mean | 0.0100 | 0.0033 | 0.0542 | 0.0284 | 75.7770 | 0.0034 | 0.0005 | 0.0022 | | 0.0019 | 0.0012 | 0.0069 | 0.0100 | 0.0001 | 0.0011 |
| STDV. | 0.0007 | 0.0012 | 0.0022 | 0.0026 | | 0.0017 | 0.0005 | 0.0028 | | 0.0023 | 0.0011 | | 0.0043 | | 0.0004 |
| Certified | 0.010 | 0.003 | 0.054 | 0.028 | | | | | | | | | (0.010) | | |
| 95% C.I. | 0.001 | 0.0009 | 0.002 | 0.002 | | | | | | | | | | | |
| Methods | X,A,O | X,D,I,O | X,I,O | X,I,O | W | A,O,D | A,O,D | | | I,D | X,D,A,O | X,A,O | X,I,O | A | X,D,I,O |
| | Methods: W = Classical, C = Combustion, F = Fusion, A = AA or GFAA, I = ICP or DCP, D = DC Arc, O = OE, X = XRF, G=GDMS | | | | | | | | | | | | | | |

The International Standards Organization (ISO) definitions, expressed in ISO Guide 30-1981- (E) list the following:

<u>Certifying Body:</u> A technically competent body (organization or firm, public or private) that issues a Reference Material Certificate. The only generally accepted certifying body in the United States is the U. S. Department of Commerce, National Institute of Standards & Technology, (NIST), Gaithersburg, MD.

<u>Reference Material (RM)</u>: A material or substance with one or more properties which are sufficiently well established to be used for calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

<u>Certified Reference Material (CRM):</u> A reference material with one or more properties whose values are certified by a technically valid procedure accompanied by or traceable to a certificate or other documentation, which is issued by a Certifying Body.

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.

<u>Methods of Analysis:</u> 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 instrument procedures.

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. Each member of the ILAP is furnished a sample pack from a specific location on the batch bar. 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.

Certified by: Diff

William D. Britt, President/General Manager Analytical Reference Materials International

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