

# Certificate of Analysis IARM 91E

CDA 932 / UNS C93200
Certified Reference Material

## Certified Values listed in wt.% with associated uncertainties

| Ag | $0.015 \pm 0.002$   | ΑI | $0.0015 \pm 0.0004$   | As | $0.008 \pm 0.002$   | Be | $0.0005 \pm 0.0004$ |
|----|---------------------|----|-----------------------|----|---------------------|----|---------------------|
| Bi | $0.109 \pm 0.009$   | С  | $0.003 \pm 0.001$     | Cd | $0.0011 \pm 0.0004$ | Co | $0.0024 \pm 0.0004$ |
| Cr | $0.0008 \pm 0.0007$ | Cu | <b>81.3</b> $\pm$ 0.2 | Fe | $0.110 \pm 0.005$   | Mn | $0.0007 \pm 0.0003$ |
| Ni | $0.300 \pm 0.006$   | Р  | $0.026 \pm 0.001$     | Pb | $7.59 \pm 0.05$     | S  | $0.028 \pm 0.002$   |
| Sb | $0.168 \pm 0.004$   | Se | $0.004 \pm 0.001$     | Si | $0.0021 \pm 0.0005$ | Sn | $6.69 \pm 0.05$     |
| Zn | <b>3.68</b> + 0.05  |    |                       |    |                     |    |                     |

## Indicative Values listed in ppm

| B (5) | Mg (<50) | N (4) | Nb (<8) | O (20) | Te (<400) |
|-------|----------|-------|---------|--------|-----------|
|-------|----------|-------|---------|--------|-----------|

## **Description and Intended Use**

This CRM may come in the form of a solid disc or chips. The intended use of this CRM may include, but is not limited to, the calibration of instruments and the validation of analytical methods.

#### Interpretation of Data

- 1. Certified values listed 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. This material was tested using both the solid disks and chips prepared from individual sections of bar. The certified values are considered representative of the overall average composition of the material.
- 3. 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.
- 4. "Provisional Certificate of Analysis" reports values that support a fully certified reference material; it also indicates that values may be in a continued process of statistical evaluation and are subject to change.
- 5. Chips are not certified for Oxygen analysis.



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

|                     |                | •            |                |              |                  |                |         |                 |               |         |               |          |          |         |          |           |
|---------------------|----------------|--------------|----------------|--------------|------------------|----------------|---------|-----------------|---------------|---------|---------------|----------|----------|---------|----------|-----------|
|                     | Ag             | Al           | As             | В            | Be               | Bi             | С       | Cd              | Со            | Cr      | Cu            | Fe       | Mg       | Mn      | N        | Nb        |
| 1                   | 0.0114         | 0.0003       | 0.0013         | 0.0003       | 0.0001           | 0.0784         | 0.0012  | 0.0007          | 0.0015        | 0.0002  | 80.60         | 0.092    | 0.0002   | 0.00006 | 0.00009  | 0.0001    |
| 2                   | 0.0149         | 0.0004       | 0.00568        | 0.0004       | 0.0002           | 0.096          | 0.0018  | 0.0007          | 0.002         | 0.0005  | 80.9774       | 0.0956   | 0.0002   | 0.0003  | 0.0001   | 0.0005    |
| 3                   | 0.015          | 0.0005       | 0.006          | 0.0005       | 0.0002           | 0.1015         | 0.00233 | 0.00075         | 0.0021        | 0.0005  | 81.07         | 0.1008   | 0.0004   | 0.0005  | 0.0002   | 0.0008    |
| 4                   | 0.015          | 0.001        | 0.006          | 0.0006       | 0.0005           | 0.105          | 0.0033  | 0.0008          | 0.0024        | 0.0006  | 81.1491       | 0.105    | < 0.0001 | 0.0005  | 0.0006   | < 0.0001  |
| 5                   | 0.0158         | 0.0011       | 0.0062         | < 0.0004     | 0.0007           | 0.1081         | 0.0034  | 0.0009          | 0.0026        | 0.0009  | 81.154        | 0.109    | < 0.0002 | 0.0007  | 0.0008   |           |
| 6                   | 0.0162         | 0.0015       | 0.0065         | < 0.005      | 0.001            | 0.109          | 0.0034  | 0.001           | 0.0027        | 0.002   | 81.18         | 0.109792 | < 0.005  | 0.0008  |          |           |
| 7                   | 0.0171         | 0.0015       | 0.0078         |              | 0.001            | 0.109          | 0.0062  | 0.0012          | 0.0027        |         | 81.20         | 0.1104   |          | 0.001   |          |           |
| 8                   |                | 0.0017       | 0.0088         |              |                  | 0.11           |         | 0.0013          | 0.003         |         | 81.234        | 0.1115   |          | 0.001   |          |           |
| 9                   |                | 0.0018       | 0.0101         |              |                  | 0.11           |         | 0.0014          |               |         | 81.2383       | 0.1137   |          | 0.001   |          |           |
| 10                  |                | 0.0019       | 0.0108         |              |                  | 0.111          |         | 0.0027          |               |         | 81.3293       | 0.115    |          |         |          |           |
| 11                  |                | 0.002        | 0.013          |              |                  | 0.1153         |         |                 |               |         | 81.34         | 0.116    |          |         |          |           |
| 12                  |                | 0.002        | 0.0143         |              |                  | 0.12           |         |                 |               |         | 81.415        | 0.1167   |          |         |          |           |
| 13                  |                | 0.0021       |                |              |                  | 0.142          |         |                 |               |         | 81.5903       | 0.118    |          |         |          |           |
| 14                  |                | 0.003        |                |              |                  |                |         |                 |               |         | 81.9729       | 0.12     |          |         |          |           |
| 15                  |                |              |                |              |                  |                |         |                 |               |         | 82.07         |          |          |         |          |           |
| Mean                | 0.015          | 0.0015       | 0.008          | 0.0005       | 0.0005           | 0.109          | 0.003   | 0.0011          | 0.0024        | 0.0008  | 81.3          | 0.11     | 0.0003   | 0.0007  | 0.0004   | 0.0005    |
| STDV.               | 0.002          | 0.0008       | 0.004          | 0.0001       | 0.0004           | 0.01           | 0.002   | 0.0006          | 0.0005        | 0.0006  | 0.4           | 0.008    | 0.0001   | 0.0003  | 0.0003   | 0.0004    |
| Certified           | 0.015          | 0.0015       | 0.008          | (0.0005)     | 0.0005           | 0.109          | 0.003   | 0.0011          | 0.0024        | 0.0008  | 81.3          | 0.110    | (<0.005) | 0.0007  | (0.0004) | (<0.0008) |
| 95% C.I.            | 0.002          | 0.0004       | 0.002          |              | 0.0004           | 0.009          | 0.001   | 0.0004          | 0.0004        | 0.0007  | 0.2           | 0.005    | , ,      | 0.0003  | ,        | ,         |
| Methods             | O,I            | X,O,I        | O,I            | O,I          | O,I              | X,O,I          | O,C     | O,I             | O,I           | O,I     | W,O,I         | X,O,I    | O,I      | O,I     | F        | O,I       |
|                     |                |              |                |              |                  |                |         |                 |               |         |               |          |          |         |          |           |
|                     | Ni             | 0            | Р              | Pb           | S                | Sb             | Se      | Si              | Sn            | Te      | Zn            |          |          |         |          |           |
| 1                   | 0.2824         | 0.001        | 0.021          | 7.468        | 0.0205           | 0.155          | 0.002   | 0.0005          | 6.55          | 0.0013  | 3.5569        |          |          |         |          |           |
| 2                   | 0.283          | 0.0011       | 0.0226         | 7.4855       | 0.021            | 0.16           | 0.0022  | 0.001           | 6.552         | 0.0033  | 3.58          |          |          |         |          |           |
| 3                   | 0.2838         | 0.00142      | 0.0249         | 7.5166       | 0.02646          | 0.165          | 0.0028  | 0.0013          | 6.5901        | 0.0047  | 3.591         |          |          |         |          |           |
| 4                   | 0.2886         | 0.0016       | 0.025          | 7.52         | 0.0265           | 0.165          | 0.0029  | 0.0015          | 6.6222        | 0.014   | 3.63          |          |          |         |          |           |
| 5                   | 0.295          | 0.0031       | 0.026          | 7.54         | 0.0276           | 0.1666         | 0.003   | 0.0015          | 6.63          | 0.0328  | 3.6345        |          |          |         |          |           |
| 6                   | 0.298238       |              | 0.0261         | 7.584        | 0.028            | 0.1672         | 0.0033  | 0.002           | 6.64          | <0.0001 | 3.636         |          |          |         |          |           |
| 7                   | 0.299          |              | 0.0265         | 7.5976       | 0.029            | 0.16775        | 0.0034  | 0.002           | 6.70          |         | 3.65          |          |          |         |          |           |
| 8                   | 0.2995         |              | 0.027          | 7.605        | 0.03             | 0.168          | 0.0035  | 0.0022          | 6.7118        |         | 3.66          |          |          |         |          |           |
| 9                   | 0.301          |              | 0.027          | 7.65         | 0.03             | 0.169          | 0.005   | 0.0022          | 6.72          |         | 3.6734        |          |          |         |          |           |
| 10                  | 0.302          |              | 0.027          | 7.664        | 0.031            | 0.171          | 0.0069  | 0.0023          | 6.72          |         | 3.72          |          |          |         |          |           |
| 11                  | 0.307          |              | 0.027          | 7.6675       | 0.031            | 0.174          |         | 0.0028          | 6.7264        |         | 3.7362        |          |          |         |          |           |
| 12                  | 0.308          |              | 0.0274         | 7.69         | 0.0311           | 0.177          |         | 0.0032          | 6.742         |         | 3.784         |          |          |         |          |           |
| 13                  | 0.3114         |              | 0.0285         | 7.712        | 0.0316           | 0.184          |         | 0.0033          | 6.7558        |         | 3.79          |          |          |         |          |           |
| 14                  | 0.315          |              | 0.029          |              | 0.0328           |                |         | 0.0037          | 6.7956        |         | 3.849         |          |          |         |          |           |
| 15                  | 0.32           |              | 0.0298         |              |                  |                |         |                 | 6.923         |         |               |          |          |         |          |           |
| Mean                | 0.3            | 0.002        | 0.026          | 7.59         | 0.028            | 0.168          | 0.004   | 0.0021          | 6.69          | 0.01    | 3.68          |          |          |         |          |           |
| STDV.               | 0.01           | 0.0008       | 0.002          | 0.08         | 0.004            | 0.007          | 0.001   | 0.0009          | 0.1           | 0.01    | 0.09          |          |          |         |          |           |
|                     | 0.300          | (0.002)      | 0.026          | 7.59         | 0.028            | 0.168          | 0.004   | 0.0021          | 6.69          | (<0.04) | 3.68          |          |          |         |          |           |
| Certified           |                | (0.002)      |                |              |                  |                |         |                 |               | (~0.07) |               |          |          |         |          |           |
| 95% C.I.<br>Methods | 0.006<br>X O I | (0.002)<br>F | 0.001<br>X O I | 0.05<br>O.LA | 0.002<br>X O I C | 0.004<br>X O I | 0.001   | 0.0005<br>X O I | 0.05<br>X O I | 0.04)   | 0.05<br>X O I |          |          |         |          |           |



# **Participating Laboratories**

I. Schumann & Company AY Mc Donald Mfg. Co. Sipi-Metals Corp Berntsen Brass & Aluminum Foundry, Inc. Riverside Brass & Aluminum Foundry Ltd. IMR Test Labs

Atlas Pacific Corporation

Bedford, OH Dubuque, IA Chicago, IL Madison, WI New Hamburg, ON Lansing, NY Colton, CA

Colorado Metallurgical Services Laboratory Testing, Inc. Anderson Laboratories, Inc. Colonial Metals Co. Concast Metal Products Co. Ingot Metal Co. Ltd.

Denver CO Hatfield, PA Greendale, WI Columbia, PA Mars PA Weston, ON

# Traceability

Members of the "Inter-Laboratory Analysis Program" (ILAP) validate test methods and instrument performance utilizing SRMs, CRMs, and RMs produced by recognized Certifying Bodies. The specific SRMs, CRMs, and RMs applicable to the material covered by this certificate are:

| ALPHA AR147  | BAM 376      | BAS 50.01-4  | BAS 54.03-4  | BCS 183-4    | BCS 364       | BNF C11.02-1  | BS 110A       | BS 175BE-1   |
|--------------|--------------|--------------|--------------|--------------|---------------|---------------|---------------|--------------|
| BS 314B      | BS 7132      | BS 822       | BS 836       | BS 903       | BS 903B       | BS 932A       | BS 932E       | BS 932M      |
| CC 905       | CC 932       | CC 932A      | CC 938-1     | CC922A       | CUCDB90       | IARM 266A     | IARM 72B      | IARM 77A     |
| IARM 78A     | IARM 82A     | IARM 86A     | IARM 87B     | IARM 89A     | IARM 90A      | IARM 90B      | IARM 91A      | IARM 91B     |
| IARM 91C     | IARM 92A     | IARM 93A     | IARM 94A     | LECO 501-644 | LECO 501-646  | LECO 501-929  | MBH 17866-K   | MBH 17868-S  |
| MBH 17868-Y  | MBH 17869-R  | MBH 17870-K  | MBH 17870-S  | MBH 31XB8-F  | MBH 32XLB13   | MBH 32XLB13B2 | MBH 32XLB14-A | MBH 32XLB1-G |
| MBH 32XLB2-F | MBH 32XLB5-D | MBH 32XLB7-B | MBH 32XPB23A | MBH 32XPB5-H | MBH 32XSEB5-A | MBH 32XSEB6-A | MBH 33XGM21A  | MBH 33XGM4-E |
| MBH 33XGM5-D | MBH 33XGM5L  | MBH 33XGM6-C | MBH 33XGM7-D | MBH 33XRB2A  | NIST 124C     | NIST 3101A    | NIST 3103A    | NIST 3105A   |
| NIST 3107    | NIST 3108    | NIST 3112A   | NIST 3113    | NIST 3131A   | NIST 3132     | NIST 3137     | NIST 3149     | NIST 3150    |
| NIST 3151    | NIST 3156    | NIST 3162A   | NIST 3169    | RC 11/20     | RC 11/4       | RC 12/12      |               |              |

# Homogeneity and Uncertainty

"Uncertainty" values, as reported adjacent to certified concentration values, are based on a 95% Confidence Interval. These estimated uncertainties include the combined effects of method imprecision, material inhomogeneity, and any bias between methods. Homogeneity data from experimental XRF results are reflected in both the overall statistics and certified data. Homogeneity samples are selected by a systematic sampling procedure. The number of samples may be determined by equation 1, where N produced is the number of units produced and N<sub>min</sub> is the number of samples used for homogeneity testing. These samples are arranged in a simple randomized design such that each sample is analyzed multiple times by XRF. Homogeneity is also determined within sample using an applied version of ASTM E826. A single factor ANOVA is used to calculated uncertainty due to inhomogeneity (U<sub>hom</sub>). Uncertainty of the material is calculated by equation 2, where H=U<sub>horn</sub>, S= Standard deviation, t= t-value at 95% CI, and n= number of observations

1. 
$$N_{min} = \max(10, \sqrt[3]{N_{prod}})$$
 2.  $U_{CRM} = \frac{\sqrt{H^2 + S^2}}{\sqrt{n}} * t$ 

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

Certifying Body: Any technically competent body (organization or firm, public or private) that issues a reference material certificate with the information detailed in ISO Guide 31. The only generally accepted certifying body in the United States for primary standards or 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, with one or more property values that 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.

Certified Reference Material (CRM): Reference material, accompanied by a certificate, with one or more property values 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.

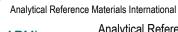
Inter-Laboratory Analysis Program (ILAP): ASTM Standard E691-87 applies to inter-laboratory studies to "Determine the Precision of a Single Test Method", but also outlines a well thought out and logical plan for conducting an inter laboratory program involving multiple analytical techniques. Therefore, the guidelines established in ASTM E691-87 were applied to all aspects of this inter laboratory program, including the protocols for planning, handling, analysis and treatment of resulting data.

Methods of Analysis: The "Inter Laboratory Analysis Program" analyzes a wide variety of materials, and as a result, no single analytical method would provide optimum analytical results. Therefore, a combination of ASTM Standard Methods for classical wet chemistry, ICP, AA, Optical Emission, X-Ray spectrometric, and other accepted methods were used to produce analytical data. Carbon, Sulfur, Nitrogen, and Oxygen results were supplied from 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 on 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. The minimum sample size for chips should be individually evaluated based on the analytical technique used; this would typically be greater than 0.1 grams. The material should be stored in a cool, dry location when not in use. Chips are not to be used for Oxygen analysis.

Selection of Materials: A "batch" or "series" is defined as a continuous length of bar produced from a single heat. The majority of IARM materials are in wrought condition; other methods of manufacture are utilized if necessary. ILAP samples are removed from equal sections from the total length of the bar. A portion of each section is converted to chips and a 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.



David Coler, General Manager