



# Certificate of Analysis

## IARM 306B

AISI M-50 / UNS T11350

### Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

<b>Al</b>	<b>0.082 ± 0.004</b>	<b>As</b>	<b>0.0040 ± 0.0009</b>	<b>B</b>	<b>0.0007 ± 0.0005</b>	<b>C</b>	<b>0.837 ± 0.005</b>
<b>Co</b>	<b>0.0100 ± 0.0005</b>	<b>Cr</b>	<b>4.12 ± 0.01</b>	<b>Cu</b>	<b>0.058 ± 0.002</b>	<b>Mn</b>	<b>0.240 ± 0.006</b>
<b>Mo</b>	<b>4.23 ± 0.04</b>	<b>N</b>	<b>0.0050 ± 0.0003</b>	<b>Nb</b>	<b>0.005 ± 0.002</b>	<b>Ni</b>	<b>0.095 ± 0.003</b>
<b>O</b>	<b>0.0009 ± 0.0004</b>	<b>P</b>	<b>0.0061 ± 0.0007</b>	<b>S</b>	<b>0.0011 ± 0.0004</b>	<b>Si</b>	<b>0.213 ± 0.009</b>
<b>Sn</b>	<b>0.0042 ± 0.0009</b>	<b>Ti</b>	<b>0.0016 ± 0.0004</b>	<b>V</b>	<b>0.98 ± 0.01</b>	<b>W</b>	<b>0.009 ± 0.003</b>
<b>Zr</b>	<b>0.0014 ± 0.0007</b>						

Indicative Values listed in ppm

Bi (<1)	Ca (10)	Mg (<20)	Pb (8)	Sb (20)	Se (<20)	Ta (<110)
Zn (<550)						

#### 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.



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

	Al	As	B	Bi	C	Ca	Co	Cr	Cu	Mg	Mn	Mo	N	Nb	Ni	O
1	0.0709	0.002	0.0001	<0.0001	0.824	0.00016	0.0089	4.075	0.0515	0.0001	0.2255	4.147	0.0043	0.0005	0.0865	0.00012
2	0.074	0.003	0.00025		0.826	0.0003	0.0092	4.087	0.0537	0.0003	0.2281	4.1766	0.0045	0.0009	0.09	0.00014
3	0.0747	0.0032	0.00054		0.8265	0.0014	0.0094	4.10	0.056	0.0017	0.231	4.1819	0.0048	0.0019	0.0903	0.0004
4	0.079	0.0034	0.0006		0.829	0.002	0.0094	4.102	0.0568	<0.0001	0.2321	4.186	0.0048	0.0051	0.093	0.0004
5	0.08	0.0042	0.0006		0.8306	0.0023	0.0098	4.105	0.057		0.236	4.1986	0.0049	0.006	0.094	0.0004
6	0.0802	0.0043	0.0007		0.8334	<0.0010	0.01	4.1062	0.0571		0.2393	4.209	0.0049	0.0065	0.095	0.0005
7	0.081	0.0045	0.0014		0.8366		0.01	4.1197	0.0572		0.24	4.231	0.0049	0.0068	0.095	0.0008
8	0.0829	0.0053	0.0018		0.838		0.01	4.127	0.058		0.241	4.24	0.0049	0.007	0.096	0.0008
9	0.083	0.0059			0.84		0.0101	4.1278	0.058		0.241	4.255	0.0051	0.0071	0.0963	0.00145
10	0.085				0.843		0.01048	4.13	0.06		0.241	4.26	0.0055	0.008	0.0988	0.0015
11	0.087				0.8434		0.0106	4.131	0.06		0.242	4.273	0.00555	0.0099	0.0992	0.0017
12	0.0879				0.84473		0.012	4.135	0.0617		0.243	4.308	0.006		0.10	0.0021
13	0.095				0.849			4.138	0.065		0.2544	4.363			0.10	
14					0.8525			4.14	0.0667							
15																
Mean	0.082	0.004	0.0007		0.837	0.001	0.01	4.12	0.058	0.001	0.24	4.23	0.005	0.005	0.095	0.0009
STDV.	0.006	0.001	0.0006		0.009	0.001	0.0008	0.02	0.004	0.0009	0.01	0.06	0.0005	0.003	0.004	0.0007
Certified	0.082	0.0040	0.0007	<0.0001	0.837	0.001	0.0100	4.12	0.058	<0.002	0.240	4.23	0.0050	0.005	0.095	0.0009
95% C.I.	0.004	0.0009	0.0005		0.005		0.0005	0.01	0.002		0.006	0.04	0.0003	0.002	0.003	0.0004
Methods	X,O,IM,I,G	X,O,IM,I	O,IM,I	O,IM	O,C	O,IM,I	X,O,IM,I,G	X,O,I,G	X,O,IM,I,G	IM,I	X,O,IM,I,G	X,O,I,G	O,F	X,O,IM,I	X,O,IM,I,G	F

	P	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr			
1	0.005	0.00001	0.0002	0.0006	0.00116	0.189	0.0013	0.001	0.0004	0.943	0.0045	0.0007	0.0002			
2	0.005	0.0001	0.0004	0.001	<0.0003	0.1934	0.0033	0.0069	0.001	0.962	0.0057	0.0023	0.0003			
3	0.0052	0.0006	0.0006	0.0014		0.1944	0.0036	0.011	0.0012	0.965	0.0063	0.05312	0.0007			
4	0.0053	0.0011	0.0007	0.002		0.2025	0.004	<0.0001	0.0013	0.969	0.0069	<0.0005	0.0015			
5	0.0054	0.0011	0.000782	0.0034		0.204	0.004		0.0013	0.971	0.007		0.0017			
6	0.0056	0.002	0.001	0.0053		0.21	0.0041		0.0014	0.978	0.008		0.0019			
7	0.0061		0.00137			0.214	0.0044		0.0019	0.9794	0.0088		0.002			
8	0.0067		0.0014			0.214	0.0048		0.002	0.9807	0.01		0.00268			
9	0.007		0.0015			0.214	0.0055		0.0021	0.9812	0.0109					
10	0.0072		0.0017			0.218	0.0056		0.0024	0.9877	0.015					
11	0.0072		0.0018			0.2195	0.006		0.0024	0.99	0.0173					
12	0.008		0.002			0.227				0.9925						
13						0.2306				1.0036						
14						0.247				1.0069						
15																
Mean	0.0061	0.0008	0.0011	0.002		0.213	0.0042	0.01	0.0016	0.98	0.009	0.02	0.0014			
STDV.	0.001	0.0007	0.0006	0.002		0.02	0.001	0.005	0.0006	0.02	0.004	0.03	0.0009			
Certified	0.0061	0.0008	0.0011	0.002	<0.002	0.213	0.0042	<0.011	0.0016	0.98	0.009	<0.055	0.0014			
95% C.I.	0.0007	0.0009	0.0004	0.002		0.009	0.0009	0.0004	0.01	0.003	0.003		0.0007			
Methods	X,O,IM,I,G	O,IM,I	O,C	O,IM,I	IM,O	X,O,IM,I,G	X,O,IM,I	X,O,IM,I	X,O,IM,I,G	X,O,I,G	X,O,IM,I,G	O,IM,I	X,O,IM,I			

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

## Participating Laboratories

ATI Specialty Materials, Lockport  
Tata Steel Limited  
Ellwood National Steel  
Crucible Industries  
Anderson Laboratories, Inc.  
NSL Analytical Services

Lockport, NY  
Jamshedpur, India  
Irvine, PA  
Syracuse, NY  
Greendale, WI  
Cleveland, OH

Exova - Alburdis  
Laboratory Testing, Inc.  
LECO Corporation  
Cronimet Specialty Metals USA, Inc.  
Colorado Metallurgical Services  
Nucor Steel Norfolk

Los Angeles, CA  
Hatfield, PA  
St. Joseph, MI  
Wheatland, PA  
Denver, CO  
Norfolk, NE

## 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 AR657	ALPHA AR660	ALPHA AR668	ALPHA AR673	ALPHA AR873	ALPHA AR878	ALPHA AR885	ALPHA AR946	ALVM50NIL
BCS 486	BCS SS401	BCS SS405/2	BCS SS406/1	BCS SS407/2	BCS SS409/2	BCS SS452/1	BCS SS454/1	BCS SS456/1
BS SS460/1	BCS SS474	BS 2A	BS 32B	BS 32D	BS 34D	BS 49	BS 60C	BS 61C
BS 8260B	BS CSN-4	CLAX C24	CLAXTON 610	IARM 155B	IARM 209C	IARM 306A	IARM 30A	IARM 38A
IARM 42B	IARM 44A	IARM 44B	IH ALV99	JSS 1005-1	LECO 501-102	LECO 501-105	LECO 501-456	LECO 501-504
LECO 501-506	LECO 501-550	LECO 501-551	LECO 501-643	LECO 501-644	LECO 501-679	LECO 501-991	LECO 502-102	LECO 502-364
LECO 502-414	LECO 502-456	LECO 762-747	M-47	NIST 1262B	NIST 132B	NIST 1763A	NIST 1764	NIST 1766
NIST 1767	NIST 293	NIST 3101A	NIST 3102A	NIST 3103A	NIST 3107	NIST 3109A	NIST 3128	NIST 3131A
NIST 3137	NIST 3155	NIST 3161A	NIST 3162A	NIST 3163	NIST 3168A	NIST 3169	NIST 361	NIST 362
NIST 363	NIST 364	NIST 50B						

## 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  $N_{prod}$  is the number of units produced and  $N_{min}$  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 calculate uncertainty due to inhomogeneity ( $U_{hom}$ ). Uncertainty of the material is calculated by equation 2, where  $H=U_{hom}$ ,  $S$ = 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.



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