



# Certificate of Analysis

## IARM 3E

AISI 309 / UNS S30900

### Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

<b>Al</b>	<b>0.0045 ± 0.0008</b>	<b>B</b>	<b>0.0005 ± 0.0003</b>	<b>C</b>	<b>0.0553 ± 0.0009</b>	<b>Ca</b>	<b>0.0015 ± 0.0003</b>
<b>Co</b>	<b>0.120 ± 0.001</b>	<b>Cr</b>	<b>22.47 ± 0.09</b>	<b>Cu</b>	<b>0.259 ± 0.004</b>	<b>Mn</b>	<b>1.62 ± 0.01</b>
<b>Mo</b>	<b>0.215 ± 0.002</b>	<b>N</b>	<b>0.063 ± 0.002</b>	<b>Nb</b>	<b>0.014 ± 0.001</b>	<b>Ni</b>	<b>11.99 ± 0.03</b>
<b>O</b>	<b>0.0048 ± 0.0005</b>	<b>P</b>	<b>0.0259 ± 0.0005</b>	<b>Pb</b>	<b>0.0003 ± 0.0002</b>	<b>S</b>	<b>0.0009 ± 0.0001</b>
<b>Si</b>	<b>0.307 ± 0.005</b>	<b>Sn</b>	<b>0.007 ± 0.001</b>	<b>Ti</b>	<b>0.0022 ± 0.0006</b>	<b>V</b>	<b>0.126 ± 0.002</b>
<b>W</b>	<b>0.018 ± 0.001</b>	<b>Zr</b>	<b>0.0018 ± 0.0004</b>				

Indicative Values listed in ppm

As (50)	H (<20)	Mg (3)	Sb (13)	Se (<220)	Ta (50)	Zn (4)
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#### 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	C	Ca	Co	Cr	Cu	H	Mg	Mn	Mo	N	Nb	Ni	O
1	0.002	0.0025	0.0002	0.0532	0.0013	0.116	22.10	0.2442	0.0011	0.0001	1.59	0.2102	0.05885	0.011	11.877	0.004
2	0.0034	0.0045	0.0003	0.0535	0.00138	0.118	22.143	0.255		0.0001	1.593	0.211	0.05965	0.0117	11.951	0.00406
3	0.00395	0.0048	0.0003	0.05365	0.0014	0.118	22.453	0.2561		0.0001	1.5989	0.2127	0.0607	0.0129	11.964	0.0041
4	0.004	0.0053	0.00035	0.0538	0.0016	0.1181	22.462	0.2567		0.0004	1.612	0.2136	0.0609	0.0132	11.965	0.0044
5	0.004	0.006	0.0004	0.054	0.0016	0.1185	22.48	0.2571		0.0006	1.617	0.214	0.0617	0.0135	11.972	0.0044
6	0.004		0.0004	0.0545	0.002	0.1191	22.498	0.258			1.617	0.215	0.0619	0.0138	11.98	0.0045
7	0.0041		0.0005	0.054875		0.12	22.50	0.259			1.623	0.215	0.062	0.014	11.98	0.0047
8	0.0047		0.0006	0.0556		0.12	22.50	0.261			1.6281	0.216	0.0621	0.0147	12.0005	0.0053
9	0.0047		0.0007	0.056		0.1201	22.5193	0.262			1.6321	0.2169	0.063	0.0148	12.0042	0.0054
10	0.0054		0.0016	0.056		0.1203	22.54	0.263			1.635	0.217	0.0639	0.0156	12.007	0.0061
11	0.006			0.0561		0.121	22.546	0.2632			1.635	0.217	0.0658	0.0156	12.03	0.0063
12	0.0063			0.0575		0.1226	22.5804	0.264			1.64	0.217	0.0667	0.0165	12.038	
13	0.0064			0.0576		0.1227	22.59	0.2681			1.6478		0.068		12.08	
14				0.0578		0.126	22.6072									
15																
Mean	0.0045	0.005	0.0005	0.0553	0.0015	0.12	22.47	0.259		0.0003	1.62	0.215	0.063	0.014	11.99	0.0048
STDV.	0.001	0.001	0.0004	0.002	0.0003	0.003	0.2	0.006		0.0002	0.02	0.002	0.003	0.002	0.05	0.0008
Certified	0.0045	(0.005)	0.0005	0.0553	0.0015	0.120	22.47	0.259	(<0.002)	(0.0003)	1.62	0.215	0.063	0.014	11.99	0.0048
95% C.I.	0.0008		0.0003	0.0009	0.0003	0.001	0.09	0.004			0.01	0.002	0.002	0.001	0.03	0.0005
Methods	O,I,G	X,O,IM,H	O,I,G	O,C	O,I,G	X,O,I,G	X,W,O,I	X,O,I,G	F	O,IM,I	X,O,I,G	X,O,I,G	O,F	X,O,I,G	X,O,I,G	O,F

	P	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr			
1	0.0241	0.000016	0.0005	0.0007	0.00005	0.2892	0.004	0.0011	0.0011	0.12	0.015	0.0001	0.0013			
2	0.0249	0.00005	0.0006	0.0012	0.0005	0.292	0.0053	0.0035	0.0014	0.1227	0.0154	0.0002	0.0015			
3	0.025	0.0003	0.0007	0.0013	0.0013	0.3023	0.0054	0.005	0.0018	0.1241	0.0158	0.0004	0.0019			
4	0.0255	0.0003	0.00072	0.0019	0.0191	0.304	0.006	0.005	0.0019	0.1246	0.0167	0.0005	0.002			
5	0.0256	0.0005	0.0008		0.0196	0.306	0.0072	0.009	0.002	0.125	0.0172	0.0006	0.002			
6	0.0258	0.00059	0.00084		0.021	0.3075	0.0078		0.0022	0.126	0.0174		0.0022			
7	0.026		0.0009			0.3076	0.008		0.0022	0.126	0.0182					
8	0.0264		0.0009			0.308	0.0085		0.0022	0.126	0.019					
9	0.0264		0.00098			0.31	0.0087		0.0027	0.1262	0.019					
10	0.0265		0.001			0.3121	0.0088		0.004	0.1264	0.0196					
11	0.0265		0.001			0.316	0.009			0.129	0.0211					
12	0.0267		0.001			0.317	0.009			0.1309	0.022					
13	0.027		0.001			0.3178				0.131						
14			0.0016							0.132						
15																
Mean	0.0259	0.0003	0.0009	0.0013	0.01	0.307	0.007	0.005	0.0022	0.126	0.018	0.0004	0.0018			
STDV.	0.0008	0.0002	0.0003	0.0005	0.01	0.009	0.002	0.003	0.0008	0.003	0.002	0.0002	0.0003			
Certified	0.0259	0.0003	0.0009	(0.0013)	(<0.022)	0.307	0.007	(0.005)	0.0022	0.126	0.018	(0.0004)	0.0018			
95% C.I.	0.0005	0.0002	0.0001		0.01	0.005	0.001	0.0006	0.002	0.002	0.001		0.0004			
Methods	X,O,I,G	O,IM,H	O,C	X,O,IM,I	X,O,I,H	X,O,I	X,O,IM,I	X,O,I	X,O,I,G	X,O,I,G	X,O,I,G	X,O,IM,H	X,O,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

Alcoa Howmet, Research Center  
Crucible Industries  
Laboratorio Prove Materiali S. Marco srl  
Special Metals IncoTest  
Cronimet Specialty Metals USA, Inc.  
Latrobe Specialty Metals, A Carpenter Co.

Whitehall, MI  
Syracuse, NY  
Schio, Italy  
Hereford, UK  
Wheatland, PA  
Latrobe, PA

ATI Powder Metals  
Laboratory Testing, Inc.  
LECO Corporation  
Anderson Laboratories, Inc.  
TimkenSteel Corporation  
Colorado Metallurgical Services

Pittsburgh, PA  
Hatfield, PA  
St. Joseph, MI  
Greendale, WI  
Canton, OH  
Denver, CO

## 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 AR654	ALPHA AR655	ALPHA AR660	ALPHA AR662	ALPHA AR669	ALPHA AR673	ALPHA AR871	ALPHA AR872	ALPHA AR882
ALPHA AR892C	ASTM 0541	ASTM 542	ASTM 9822	BAS 401/1	BAS 401/2	BAS 402/1	BAS 403/1	BAS 404/1
BAS 405/1	BAS 406/1	BAS 407/2	BAS 408/1	BAS 409/1	BAS 410/2	BAS 465/1	BAS 466/1	BAS 470
BAS 472	BAS 473	BAS 474	BAS 475	BAS SS451	BAS SS452	BAS SS453	BAS SS454	BAS SS456
BAS SS457	BAS SS458	BAS SS459	BAS SS460	BCS 351/1	BCS 454/1	BCS 462/1	BS 82A	BS 82C
BS 82E	BS 83B	BS 83C	BS CA-1	BS CA1A	BS CA-2	BS CA-3	BS CA316-4	BS CA3A
BS CA-4	ES 252/1	ES 281/1	ES 287/1	HAS 317B	IARM 11A	IARM 16B	IARM 190A	IARM 21B
IARM 2C	IARM 316A	IARM 317A	IARM 3A	IARM 3B	IARM 3C	IARM 4B	IARM 6B	IARM 9A
IARM 9B	IARM 9C	IH R5657	JSS 168-4	JSS 169-4	JSS 170-4	JSS 171-4	JSS 190-1	JSS 191-1
JSS 192-1	JSS 193-1	JSS 194-1	JSS 195-1	JSS 650-11	JSS 653-11	JSS ST01	JSS ST02	JSS ST03
JSS ST04	JSS ST05	LECO 501-102	LECO 501-502	LECO 501-503	LECO 501-551	LECO 501-642	LECO 501-643	LECO 501-644
LECO 501-646	LECO 501-992	LECO 502-064	LECO 502-102	LECO 502-195	LECO 502-257	LECO 502-411	LECO 502-416	MBH 128X353
MBH 13X12854K	MBH 13X12855L	MBH 13X14216N	NIST 101E	NIST 101G	NIST 1261A	NIST 1262	NIST 1262B	NIST 1263
NIST 1264	NIST 16F	NIST 1754	NIST 1760	NIST 1761	NIST 1762	NIST 1763	NIST 1764	NIST 1765
NIST 1766	NIST 1767	NIST 2166	NIST 3101A	NIST 3103A	NIST 3109A	NIST 3128	NIST 3137	NIST 3155
NIST 3163	NIST 3168A	NIST 3169	NIST 339	NIST 343A	NIST 346	NIST 361	NIST 362	NIST 363
NIST 368	NIST C1154	VHG 002-0024	VHG 002-0057R	VHG 007-0054R	VHG 010-0030	VHG 012-0075R	VHG 012-0164F	VHG 104-0034R
VHG 104-0064R	VHG 109-0005	VHG 911-0090						

## 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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