



# Certificate of Analysis

## IARM 13D

AISI 440C / UNS S44004

### Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

<b>Al</b>	<b>0.006 ± 0.001</b>	<b>As</b>	<b>0.008 ± 0.002</b>	<b>B</b>	<b>0.0005 ± 0.0003</b>	<b>C</b>	<b>1.040 ± 0.004</b>
<b>Co</b>	<b>0.0212 ± 0.0006</b>	<b>Cr</b>	<b>16.36 ± 0.03</b>	<b>Cu</b>	<b>0.184 ± 0.006</b>	<b>Mn</b>	<b>0.697 ± 0.009</b>
<b>Mo</b>	<b>0.488 ± 0.006</b>	<b>N</b>	<b>0.0492 ± 0.0009</b>	<b>Nb</b>	<b>0.0074 ± 0.0008</b>	<b>Ni</b>	<b>0.256 ± 0.004</b>
<b>O</b>	<b>0.0031 ± 0.0002</b>	<b>P</b>	<b>0.0195 ± 0.0006</b>	<b>S</b>	<b>0.0012 ± 0.0002</b>	<b>Sb</b>	<b>0.0027 ± 0.0009</b>
<b>Si</b>	<b>0.614 ± 0.007</b>	<b>Sn</b>	<b>0.011 ± 0.001</b>	<b>Ta</b>	<b>0.004 ± 0.003</b>	<b>Ti</b>	<b>0.0035 ± 0.0007</b>
<b>V</b>	<b>0.058 ± 0.003</b>	<b>W</b>	<b>0.046 ± 0.008</b>	<b>Zr</b>	<b>0.0017 ± 0.0006</b>		

Indicative Values listed in ppm

Ca (7)	Ce (<50)	Mg (<200)	Pb (<90)	Se (<200)	Zn (<200)
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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	Ce	Co	Cr	Cu	Mg	Mn	Mo	N	Nb	Ni	O
1	0.001	0.004	0.0001	1.0296	0.00046	<0.0001	0.019	16.30	0.171	0.0001	0.664	0.4674	0.047	0.006	0.245	0.0028
2	0.0037	0.004	0.0003	1.03	0.0006	<0.005	0.0204	16.309	0.172	0.0001	0.68	0.47	0.0472	0.0063	0.246	0.0029
3	0.0044	0.007	0.0005	1.032	0.0007		0.0207	16.31	0.174	0.0002	0.686	0.48	0.048	0.0067	0.2492	0.0029
4	0.0049	0.007	0.0006	1.0374	0.0007		0.021	16.32	0.178	0.0008	0.687	0.483	0.0485	0.007	0.25	0.003
5	0.0055	0.0089	0.0008	1.04	0.0008		0.021	16.32	0.1783	0.012	0.69	0.485	0.0492	0.007	0.25	0.0031
6	0.0055	0.0101	0.0008	1.04	<0.003		0.021	16.33	0.179		0.691	0.488	0.04969	0.007	0.25	0.0032
7	0.006	0.0103		1.0408			0.0211	16.3562	0.179		0.691	0.4917	0.0498	0.008	0.2539	0.0036
8	0.0062	0.011		1.043			0.0213	16.3715	0.18		0.6956	0.493	0.05	0.0081	0.2557	
9	0.0066			1.043			0.022	16.372	0.184		0.696	0.493	0.05	0.0086	0.256	
10	0.0072			1.045			0.022	16.39	0.1843		0.703	0.494	0.0506	0.0093	0.2565	
11	0.0073			1.047			0.022	16.407	0.1844		0.707	0.4953	0.0511		0.258	
12	0.0077			1.05			0.023	16.412	0.188		0.7095	0.4961			0.2621	
13	0.008							16.419	0.20		0.7166	0.497			0.265	
14	0.01								0.20		0.72	0.50			0.2668	
15									0.213		0.722				0.27	
Mean	0.006	0.008	0.0005	1.04	0.0007		0.0212	16.36	0.184	0.003	0.697	0.488	0.0492	0.0074	0.256	0.0031
STDV.	0.002	0.003	0.0003	0.007	0.0001		0.001	0.04	0.01	0.005	0.02	0.01	0.001	0.001	0.008	0.0003
Certified	0.006	0.008	0.0005	1.040	(0.0007)	(<0.005)	0.0212	16.36	0.184	(<0.02)	0.697	0.488	0.0492	0.0074	0.256	0.0031
95% C.I.	0.001	0.002	0.0003	0.004			0.0006	0.03	0.006		0.009	0.006	0.0009	0.0008	0.004	0.0002
Methods	X,O,I	X,O,IM	X,O,IM,I	O,C	X,O,I	O,IM	X,O,I	X,W,O,I	X,O,I	O,I,H	X,O,I	X,O,I	O,F	X,O,I	X,O,I	F

	P	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr
1	0.0172	0.0001	0.0008	0.0014	0.0001	0.588	0.0052	0.0016	0.0022	0.0474	0.015	0.0002	0.001
2	0.0174	0.0007	0.000865	0.0022	0.0001	0.595	0.0096	0.0022	0.0026	0.051	0.0243	0.0028	0.0014
3	0.019	0.0011	0.0009	0.0024	0.0002	0.5975	0.0097	0.0026	0.0028	0.056	0.029	0.0033	0.0014
4	0.019	0.0011	0.0009	0.003	0.0003	0.5975	0.01	0.003	0.003	0.0564	0.0384	0.016	0.0015
5	0.019	0.002	0.001	0.003	0.009	0.612	0.0101	0.003	0.003	0.0568	0.0455		0.0016
6	0.0194	0.0059	0.001	0.004	0.016	0.6146	0.0107	0.0094	0.003	0.058	0.0483		0.0017
7	0.0195	0.0089	0.001			0.6178	0.0108		0.0033	0.0582	0.0513		0.003
8	0.0198		0.0012			0.6193	0.011		0.0035	0.059	0.0524		
9	0.02		0.0014			0.62	0.011		0.0039	0.059	0.053		
10	0.02		0.00169			0.62	0.012		0.0042	0.0593	0.054		
11	0.02		0.0018			0.621	0.012		0.0044	0.062	0.0562		
12	0.02		0.0019			0.622	0.012		0.00602	0.062	0.058		
13	0.0203					0.624	0.0125			0.0634	0.058		
14	0.0204					0.6262				0.064	0.059		
15	0.021					0.63							
Mean	0.0195	0.003	0.0012	0.0027	0.004	0.614	0.011	0.004	0.0035	0.058	0.046	0.01	0.0017
STDV.	0.001	0.003	0.0004	0.0009	0.007	0.01	0.002	0.003	0.001	0.005	0.01	0.007	0.0006
Certified	0.0195	(<0.009)	0.0012	0.0027	(<0.02)	0.614	0.011	0.004	0.0035	0.058	0.046	(<0.02)	0.0017
95% C.I.	0.0006		0.0002	0.0009		0.007	0.001	0.003	0.0007	0.003	0.008		0.0006
Methods	X,O,I	O,I,H	O,C	O,IM,H	O,IM,I,H	X,O,I	X,O,IM,I	X,O,I	X,O,I	X,O,I	X,O,I	O,IM,H	X,O,IM

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

Exova - Burlington  
Crucible Industries  
Davis Alloys Manufacturing, LLC  
Anderson Laboratories, Inc.  
Laboratorio Prove Materiali S. Marco srl  
PM Kalco, Inc

Burlington, ON  
Syracuse, NY  
Sharpville, PA  
Greendale, WI  
Schio, Italy  
Wheatland, PA

AADFW, Inc.  
Laboratory Testing, Inc.  
SPECTRO Analytical Instruments Inc.  
Special Metals IncoTest  
Eilwood National Steel  
AK Steel, Research Center

Eules, TX  
Hatfield, PA  
Mahwah, NJ  
Hereford, UK  
Irvine, PA  
Middletown, OH

## 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:

AKS 588	AKS 603	AKS 689	AKV 2816	ALPHA AR 881	ALPHA AR654	ALPHA AR881	ALPHA AR883	ALPHA AR889
ASTM 0122	ASTM 1122	ASTM 941	ASTM 942	BRAMMER 91F	BS 156	BS 440C	BS 4951	BS 60C
BS 84J	BS 90F	BS 93C	BS 93E	BS 94C	BS 98	FR FCR3	FR FCR7	IARM 13B
IARM 13C	IARM 154A	IARM 154B	IARM 15A	IARM 15B	IARM 162C	IARM 16B	IARM 205A	IARM 205B
IARM 20G	IARM 21B	IARM 2B	IARM 2C	IARM 4B	IARM 6G	LECO 501-503	LECO 501-504	LECO 501-506
LECO 501-952	LECO 501-991	LECO 501-992	LECO 502-102	LECO 502-106	LECO 502-416	LECO 502-494	MBH 13X44004A	MBH 18X312A
NIST 2168	NIST 3107	NIST 3109A	NIST 3137	NIST 3149	NIST 3155	NIST 3162A	NIST 3163	NIST 3165
NIST 3168A	NIST 3169	NIST 337	NIST 361	NIST 363	NIST 73B	V2867		

## 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.



David Coler, General Manager

Analytical Reference Materials International



Analytical Reference Materials International • 276 Abby Road • Manchester, NH 03103  
Telephone (603) 935-4100 • Fax (603) 935-4101 • www.ARMl.com • ARMI@LGCgroup.com



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