Certificate of Analysis

IARM 320A

AISI M-35 / UNS T11335

Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

Al	**0.023** ± 0.004	**As**	**0.013** ± 0.005	**B**	**0.0011** ± 0.0004	**C**	**0.93** ± 0.01
Co	**4.90** ± 0.03	**Cr**	**4.22** ± 0.05	**Cu**	**0.091** ± 0.004	**Mn**	**0.33** ± 0.01
Mo	**4.79** ± 0.06	**N**	**0.014** ± 0.002	**Nb**	**0.015** ± 0.007	**Ni**	**0.204** ± 0.007
O	**0.0021** ± 0.0008	**P**	**0.021** ± 0.002	**S**	**0.0015** ± 0.0008	**Si**	**0.36** ± 0.01
Sn	**0.008** ± 0.001	**Ti**	**0.0032** ± 0.0008	**V**	**1.76** ± 0.04	**W**	**6.01** ± 0.07

Indicative Values listed in ppm

Bi (<1)	Ca (<30)	Mg (<30)	Pb (<70)	Sb (<11)	Se (<1)	Ta (<20)
Zn (<10)	Zr (30)					

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	0.0077	0.0046	0.00028	0.000002	0.8836	0.0026	4.84	4.15	0.082	0.0003	0.308	4.642	0.0085	0.005	0.18	0.001
2	0.013	0.00644	0.0005	0.00001	0.884		4.85	4.1567	0.084	0.0022	0.3091	4.7174	0.01	0.0076	0.187	0.0011
3	0.0191	0.009	0.001		0.93		4.85	4.169	0.0852	0.0022	0.314	4.72	0.011	0.0094	0.196	0.0013
4	0.02	0.0095	0.0012		0.9328		4.86	4.17	0.086		0.316	4.72	0.0142	0.0116	0.1968	0.0021
5	0.02	0.0111	0.0014		0.9368		4.9015	4.18	0.088		0.32	4.75	0.0145	0.015	0.20	0.0024
6	0.0207	0.0132	0.00147		0.93795		4.913	4.20	0.0889		0.3234	4.758	0.0147	0.0165	0.20	0.0026
7	0.024	0.015	0.00155		0.9385		4.921	4.208	0.0905		0.325	4.81	0.0147	0.0166	0.202	0.003
8	0.024	0.0202	0.0016		0.94		4.9355	4.223	0.091		0.325	4.812	0.0151	0.0241	0.206	0.0036
9	0.027	0.0238			0.94		4.94	4.26	0.0939		0.334	4.85	0.01513	0.032	0.2069	
10	0.028				0.9461		4.9453	4.26	0.096		0.337	4.875	0.0153		0.208	
11	0.029				0.949			4.4342	0.0979		0.3389	4.90	0.0156		0.211	
12	0.0301				0.952				0.098		0.3432	4.955	0.0158		0.2116	
13	0.0318				0.953				0.10		0.3645		0.0176		0.222	
14					0.954										0.228	
15																
Mean	0.023	0.013	0.0011	0.00001	0.93		4.9	4.22	0.091	0.002	0.33	4.79	0.014	0.015	0.204	0.0021
STDV.	0.007	0.006	0.0005	0.000006	0.02		0.04	0.08	0.006	0.001	0.02	0.09	0.003	0.008	0.01	0.0009
Certified	**0.023**	**0.013**	**0.0011**	**(<0.0001)**	**0.93**	**(<0.003)**	**4.90**	**4.22**	**0.091**	**(<0.003)**	**0.33**	**4.79**	**0.014**	**0.015**	**0.204**	**0.0021**
95% C.I.	0.004	0.005	0.0004		0.01		0.03	0.05	0.004		0.01	0.06	0.002	0.007	0.007	0.0008
Methods	X,O,I,G	X,O,IM,I	O,I	IM,I	O,C	I	X,O,I,G	X,W,O,I,G	X,O,I,G	IM,I	X,O,I,G	X,O,I,G	O,F	X,O,I,G	X,O,I,G	F

	P	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr			
1	0.0158	0.000018	0.0001	0.001	0.00001	0.32	0.0049	0.0007	0.0011	1.61	5.8285	0.0003	0.0008			
2	0.0168	0.0002	0.00026	0.0011	0.000057	0.329	0.005	0.001	0.002	1.6668	5.911		0.001			
3	0.019	0.0067	0.00035			0.355	0.00662	0.002	0.0021	1.726	5.9222		0.0014			
4	0.0191		0.0004			0.355	0.007		0.0021	1.736	5.93		0.0042			
5	0.02		0.001			0.355	0.0078		0.0022	1.74	5.9483		0.0057			
6	0.02		0.001			0.3559	0.0085		0.003	1.77	6.012					
7	0.0207		0.0013			0.3607	0.0085		0.0034	1.772	6.02					
8	0.0207		0.0015			0.362	0.0089		0.0035	1.773	6.021					
9	0.0207		0.0016			0.368	0.0091		0.0039	1.78	6.082					
10	0.0222		0.002			0.3793	0.01		0.0041	1.79	6.145					
11	0.023		0.0028			0.3837	0.0118		0.0044	1.8104	6.15					
12	0.0247		0.0034			0.384			0.005	1.8133	6.20					
13	0.025		0.0042			0.3951			0.005	1.83						
14																
15																
Mean	0.021	0.002	0.0015	0.0011	0.00003	0.36	0.008	0.001	0.0032	1.76	6.01		0.003			
STDV.	0.003	0.004	0.001	0.00007	0.00003	0.02	0.002	0.0007	0.001	0.06	0.1		0.002			
Certified	**0.021**	**(<0.007)**	**0.0015**	**(0.0011)**	**(<0.0001)**	**0.36**	**0.008**	**(<0.003)**	**0.0032**	**1.76**	**6.01**	**(<0.001)**	**(0.003)**			
95% C.I.	0.002		0.0008			0.01	0.001		0.0008	0.04	0.07					
Methods	X,O,I	O,IM,I	O,C	IM	IM	X,W,O,I,G	O,IM,I	O,I	X,O,I,G	X,O,I,G	X,O,I,G	IM,I	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

MetalTek International, Inc.	Waukesha, WI	Exova - Portland	Portland, OR
Latrobe Specialty Metals, A Carpenter Co.	Latrobe, PA	Laboratory Testing, Inc.	Hatfield, PA
Crucible Industries	Syracuse, NY	LECO Corporation	St. Joseph, MI
TMK IPSCO	Koppel, PA	ATI Powder Metals	Pittsburgh, PA
Anderson Laboratories, Inc.	Greendale, WI	Colorado Metallurgical Services	Denver, CO
Special Metals IncoTest	Hereford, UK	RG-Steel	Mingo Junction, 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:

ALPHA AR660	ALPHA AR668	ALPHA AR669	ALPHA AR875	ALPHA AR881	BAS SS 483-1	BAS SS 484-1	BAS SS 486	BAS SS 486-1
BAS SS 487-1	BCS 351	BCS 407	BCS 454/1	BCS 462/1	BCS 485	BCS 486	BS 10V	BS 32B
BS 32C	BS 33E	BS 34C	BS 36C	BS 37C	BS 37E	BS 38B	BS 3941	BS 39B
BS 40	BS 46	BS 50B	BS 51C	HAS 223A	HAS 276A	IARM 306A	IARM 32C	IARM 39A
IARM 40A	IARM 41A	IARM 42A	IARM 43A	IARM 44A	IARM 44B	IARM 44C	IARM 45A	IARM 46A
IARM 47A	IARM 48B	IARM 49C	LECO 501-505	LECO 501-506	LECO 501-551	LECO 501-643	LECO 502-102	LECO 502-257
NIST 1157	NIST 1269	NIST 1282	NIST 132A	NIST 132B	NIST 134	NIST 1760	NIST 1761	NIST 1762
NIST 1763	NIST 1764	NIST 1765	NIST 1766	NIST 1767	NIST 1768	NIST 3101A	NIST 3103A	NIST 3106
NIST 3109A	NIST 3113	NIST 3128	NIST 3131A	NIST 3137	NIST 3155	NIST 3161A	NIST 3162A	NIST 3168A
NIST 3169	NIST 361	NIST 362	NIST 363	NIST 364	NIST3107	R3386	R5657	RD 3310
SS 483-1	SS 486/1	SS 487-1						

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 calculated 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