Certificate of Analysis

IARM 10D

AISI 416 / UNS S41600

Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

Al	**0.0027** ± 0.0008	**As**	**0.007** ± 0.003	**B**	**0.002** ± 0.001	**C**	**0.110** ± 0.002
Co	**0.0187** ± 0.0009	**Cr**	**12.42** ± 0.07	**Cu**	**0.192** ± 0.003	**Mn**	**1.11** ± 0.02
Mo	**0.148** ± 0.003	**N**	**0.0241** ± 0.0008	**Nb**	**0.0027** ± 0.0005	**Ni**	**0.291** ± 0.004
O	**0.005** ± 0.001	**P**	**0.0178** ± 0.0005	**S**	**0.334** ± 0.008	**Si**	**0.475** ± 0.009
Sn	**0.010** ± 0.001	**Ti**	**0.0015** ± 0.0005	**V**	**0.051** ± 0.001	**W**	**0.005** ± 0.002

Indicative Values listed in ppm

Ca (11) Mg (<90) Pb (3) Sb (22) Se (<200) Ta (<50) Zn (60)

Zr (<50)

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	Mg	Mn	Mo	N	Nb	Ni	O	P
1	0.001	0.0028	0.0003	0.106	0.00091	0.0158	12.237	0.182	0.0001	1.08	0.136	0.0228	0.0013	0.275	0.0025	0.0166
2	0.0012	0.003	0.0004	0.108	0.00092	0.017	12.3245	0.187	0.0008	1.082	0.1417	0.02299	0.0019	0.2783	0.0046	0.017
3	0.0013	0.0054	0.0004	0.1086	0.0011	0.0174	12.33	0.189	0.0081	1.0888	0.145	0.023	0.0024	0.281	0.0048	0.017
4	0.0017	0.0057	0.00076	0.109	0.0011	0.0182	12.342	0.1894	0.0082	1.09	0.147	0.023	0.0027	0.288	0.0049	0.017
5	0.002	0.0062	0.0017	0.109	0.0013	0.0182	12.353	0.19	0.0087	1.0975	0.147	0.0235	0.0028	0.289	0.0051	0.0174
6	0.0023	0.0063	0.0018	0.1093	<0.001	0.0182	12.362	0.19		1.10	0.1479	0.024	0.003	0.289	0.0057	0.0175
7	0.0025	0.0108	0.0027	0.1094	<0.005	0.0185	12.364	0.19		1.1043	0.148	0.024	0.0033	0.29	0.006	0.0177
8	0.0033	0.012	0.0032	0.11		0.019	12.37	0.192		1.105	0.149	0.024	0.0033	0.291	0.0064	0.0178
9	0.00363		0.0037	0.1103		0.019	12.39	0.195		1.109	0.15	0.0243	0.0034	0.294		0.018
10	0.0042			0.112		0.0192	12.44	0.1954		1.114	0.15	0.0258		0.296		0.018
11	0.0042			0.11218		0.0194	12.47	0.196		1.119	0.151	0.026		0.296		0.0183
12	0.0048			0.115		0.02	12.522	0.197		1.12	0.1512	0.0264		0.2972		0.0185
13				0.1156		0.0204	12.5525	0.205		1.14	0.1548			0.298		0.0186
14						0.022	12.57			1.178	0.156			0.2993		0.0197
15							12.72			1.195				0.30		
Mean	0.0027	0.007	0.002	0.11	0.0011	0.0187	12.42	0.192	0.005	1.11	0.148	0.0241	0.0027	0.291	0.005	0.0178
STDV.	0.001	0.003	0.001	0.003	0.0002	0.002	0.1	0.006	0.004	0.03	0.005	0.001	0.0007	0.008	0.001	0.0008
Certified	**0.0027**	**0.007**	**0.002**	**0.110**	**(0.0011)**	**0.0187**	**12.42**	**0.192**	**(<0.009)**	**1.11**	**0.148**	**0.0241**	**0.0027**	**0.291**	**0.005**	**0.0178**
95% C.I.	0.0008	0.003	0.001	0.002		0.0009	0.07	0.003		0.02	0.003	0.0008	0.0005	0.004	0.001	0.0005
Methods	X,O,I	X,O,IM,I,H	X,O,I	O,C	X,O,I	X,O,I	X,O,I	X,O,I	O,IM,H	X,O,I	X,O,I	O,F,C	X,O,I	X,O,I	O,F	X,O,I

	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr				
1	0.0001	0.3107	0.0015	0.0001	0.443	0.006	0.001	0.0002	0.049	0.0016	0.0014	0.0004				
2	0.0002	0.3121	0.0016	0.0002	0.45	0.006	0.002	0.001	0.0497	0.0016	0.0042	0.001				
3	0.0002	0.3174	0.0021	0.0004	0.4646	0.0098	<0.0001	0.001	0.0498	0.0016	0.0049	0.0011				
4	0.0003	0.319	0.0023	0.0005	0.4668	0.0099	<0.0010	0.0011	0.05	0.003	0.013	0.0011				
5	0.00071	0.3328	0.0034	0.0144	0.4729	0.01	<0.003	0.0011	0.05	0.0036	<0.005	0.0023				
6	<0.001	0.3363	<0.004	0.021	0.473	0.01	<0.003	0.0013	0.0505	0.005		<0.002				
7	<0.003	0.3373	<0.005		0.4736	0.01	<0.005	0.0015	0.0509	0.0058		<0.005				
8	<0.003	0.3397			0.476	0.0102	<0.005	0.00155	0.051	0.006						
9		0.34			0.477	0.0102	<0.010	0.0018	0.051	0.0061						
10		0.34			0.481	0.0107		0.0026	0.0521	0.009						
11		0.344			0.481	0.0109		0.00293	0.0522	0.01						
12		0.3449			0.481	0.011			0.0538							
13		0.346			0.4935	0.0111			0.0552							
14		0.355			0.497	0.012										
15					0.498											
Mean	0.0003	0.334	0.0022	0.01	0.475	0.01	0.002	0.0015	0.051	0.005	0.006	0.0012				
STDV.	0.0002	0.01	0.0008	0.009	0.02	0.002	0.0007	0.0008	0.002	0.003	0.005	0.0007				
Certified	**(0.0003)**	**0.334**	**(0.0022)**	**(<0.02)**	**0.475**	**0.010**	**(<0.005)**	**0.0015**	**0.051**	**0.005**	**(0.006)**	**(<0.005)**				
95% C.I.		0.008			0.009	0.001		0.0005	0.001	0.002						
Methods	X,O,IM,H	X,O,C	X,O,IM,I,H	X,O,IM,I,H	X,O,I	X,O,IM,I	X,O,I	X,O,IM,I	X,O,I	X,O,I	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

Exova - Burlington	Burlington, ON	AADFW, Inc.	Euless, TX
Crucible Industries	Syracuse, NY	Laboratory Testing, Inc.	Hatfield, PA
Davis Alloys Manufacturing, LLC	Sharpsville, PA	SPECTRO Analytical Instruments Inc.	Mahwah, NJ
Anderson Laboratories, Inc.	Greendale, WI	Special Metals IncoTest	Hereford, UK
Laboratorio Prove Materiali S. Marco srl	Schio, Italy	Ellwood National Steel	Irvine, PA
IMR Test Labs	Lansing, NY	AK Steel, Research Center	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:

ALPHA AR1650	ALPHA AR511	ALPHA AR654	ALPHA AR883	ASTM 0121	ASTM 1121	ASTM 1122	ASTM 122	ASTM 9731
BCS SS472	BS 156	BS 60C	BS 84J	BS 8620B	BS 89C	BS 89E	BS 90C	BS 90D
BS 90F	BS 93E	BS 94C	BS 98	BS T416	EURONORM 286-1	IARM 10A	IARM 10B	IARM 10C
IARM 13C	IARM 154B	IARM 16B	IARM 182B	IARM 1B	IARM 205A	IARM 205B	IARM 205C	IARM 21B
IARM 2B	IARM 2C	IARM 2F	IARM 302A	IARM 35J	IARM 4B	IARM 9C	IH AK V2816	IH AK V2867
IH AKS 584	IH AKS 585	IH AKS 598	IH AKS 669	LECO 501-503	LECO 501-551	LECO 501-645	LECO 501-646	LECO 501-676
LECO 501-991	LECO 501-992	LECO 502-106	LECO 502-416	LECO 502-449	LECO 502-494	MBH 13X41600A	MBH 13X44004A	MBH 316
NIST 1223	NIST 1295	NIST 1296	NIST 129A	NIST 133A	NIST 3109A	NIST 3131A	NIST 3149	NIST 3155
NIST 3161	NIST 3163	NIST 3168A	NIST 3169	NIST 361	NIST 363	NIST 36B	NIST 73B	SS4951
SS4952								

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

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