Certificate of Analysis

IARM 169B

Low Alloy Steel - CLA6

Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

Al	**0.36** ± 0.01	**B**	**0.0003** ± 0.0001	**C**	**0.232** ± 0.004	**Co**	**0.003** ± 0.001
Cr	**0.010** ± 0.001	**Cu**	**0.005** ± 0.001	**Mn**	**0.75** ± 0.01	**Mo**	**0.004** ± 0.002
Nb	**0.004** ± 0.002	**Ni**	**0.010** ± 0.002	**P**	**0.004** ± 0.001	**S**	**0.004** ± 0.003
Si	**0.32** ± 0.04	**Sn**	**0.002** ± 0.001	**Ti**	**0.23** ± 0.02	**V**	**0.002** ± 0.001
W	**0.003** ± 0.002						

Indicative Values listed in ppm

As	(20)	Bi	(<110)	Ca	(<60)	Mg	(<50)	N	(40)	O	(40)	Pb	(10)
Sb	(9)	Se	(<50)	Ta	(<80)	Zn	(<10)	Zr	(40)				

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	0.341	0.001	0.00018	0.011	0.223	0.002	0.001	0.007	0.003	<0.001	0.707	0.001	0.00204	0.002	0.0062	0.00138
2	0.34103	0.00112	0.0002		0.2235	0.00597	0.0026	0.0089	0.0038	<0.005	0.734	0.001	0.003	0.00229	0.0068	0.0045
3	0.349	0.002	0.00024		0.225		0.00283	0.009	0.0039		0.737	0.0013	0.0034	0.0024	0.008	0.0055
4	0.35	0.0029	0.00034		0.233		0.0029	0.009	0.0044		0.738	0.0018	0.006	0.003	0.0088	<0.005
5	0.355	<0.005	0.00037		0.2331		0.004	0.01	0.0048		0.74	0.003	<0.005	0.0042	0.009	
6	0.355		0.0004		0.2332		0.005	0.0108	0.006		0.745	0.0051		0.005	0.01	
7	0.3567				0.2338			0.0109	0.0062		0.748	0.0051		0.0077	0.01	
8	0.358				0.236			0.011			0.75	0.006			0.0101	
9	0.376				0.2375			0.011			0.751	0.006			0.011	
10	0.3769				0.238			0.012			0.7511	0.01			0.013	
11	0.384							0.0124			0.7553				0.014	
12											0.785					
13																
14																
15																
Mean	0.36	0.0020	0.0003		0.232	0.004	0.003	0.01	0.005		0.75	0.004	0.004	0.004	0.01	0.004
STDV.	0.01	0.0009	0.00009		0.006	0.003	0.001	0.002	0.001		0.02	0.003	0.002	0.002	0.002	0.002
Certified	**0.36**	**(0.002)**	**0.0003**	**(<0.011)**	**0.232**	**(<0.006)**	**0.003**	**0.010**	**0.005**	**(<0.005)**	**0.75**	**0.004**	**(0.004)**	**0.004**	**0.010**	**(0.004)**
95% C.I.	0.01		0.0001		0.004		0.001	0.001	0.001		0.01	0.002		0.002	0.002	
Methods	X,O,I	X,O,I	O,I	O	O,C	O	X,O,I	X,O,I	X,O,I	O	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	0.00231	0.0002	0.001	0.00086	<0.001	0.183	0.00032	0.005	0.193	0.0005	0.00095	0.0004	0.00167			
2	0.003	0.00087	0.001	0.001	<0.005	0.265	0.00115	<0.001	0.208	0.00096	0.0014		0.002			
3	0.003	0.001	0.002	<0.004		0.3095	0.0017	<0.001	0.212	0.001	0.002		0.002			
4	0.0032	<0.001	0.003			0.3136	0.002	<0.008	0.22	0.001	0.002		0.0022			
5	0.0033	<0.001	0.0038			0.316	0.0027		0.223	0.001	0.004		0.01			
6	0.0035		0.00394			0.32	0.003		0.2271	0.002	0.006					
7	0.004		0.0042			0.321			0.23786	0.00318						
8	0.004		0.0044			0.321			0.245	0.005						
9	0.006		0.0074			0.322			0.285							
10	0.0066		0.0131			0.331										
11	0.008					0.3611										
12						0.4251										
13																
14																
15																
Mean	0.004	0.001	0.004	0.0009		0.32	0.002		0.23	0.002	0.003		0.004			
STDV.	0.002	0.0004	0.004	0.0001		0.06	0.001		0.03	0.002	0.002		0.004			
Certified	**0.004**	**(0.001)**	**0.004**	**(0.0009)**	**(<0.005)**	**0.32**	**0.002**	**(<0.008)**	**0.23**	**0.002**	**0.003**	**(<0.001)**	**(0.004)**			
95% C.I.	0.001		0.003			0.04	0.001		0.02	0.001	0.002					
Methods	X,O,I	O	X,O,C	X,O	X,O	X,O,I	X,O,I	X,O	X,O,I	X,O,I	X,O,I	X,O	X,O			

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

Anderson Laboratories, Inc.	Greendale, WI	Cronimet Specialty Metals USA, Inc.	Wheatland, PA
Davis Alloys Manufacturing, LLC	Sharpsville, PA	Exova - Burlington	Burlington, ON
Exova - Gary	Gary, IN	Laboratorio Prove Materiali S. Marco srl	Schio, Italy
Laboratory Testing, Inc.	Hatfield, PA	Nucor Steel Kankakee	Bourbonnais, IL
TimkenSteel Corporation	Canton, OH	VHG Labs	Manchester, NH

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 AR1648	ALPHA AR878	BS XCCS	IARM 252C	IARM 32D	MBH 128X 353C	NIST 132A	NIST 1765	NIST 368
ALPHA AR659	ALPHA AR883	IARM 132A	IARM 27D	IARM 35F	NIST 1262A	NIST 16F	NIST 361	SS59
ALPHA AR667	BCS 432-2	IARM 209D	IARM 28H	LECO 501-646	NIST 1263	NIST 1754	NIST 362	
ALPHA AR670	BS 50B	IARM 210D	IARM 28J	LECO 501-675	NIST 1263A	NIST 1763	NIST 363	
ALPHA AR874	BS CSN2-2	IARM 241A	IARM 31E	LECO 502-016	NIST 1264A	NIST 1764	NIST 364	

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.

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Analytical Reference Materials International

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