

Certificate of Analysis IARM 332A

CMSX-4

Certified Reference Material

Certified Values listed in wt.% with associated uncertainties

ΑI	5.79 ± 0.03	В	0.0009 ± 0.0001	C	0.0039 ± 0.0004	Co	9.45 ± 0.07	
Cr	6.3 ± 0.1	Cu	0.007 ± 0.005	Fe	0.028 ± 0.006	Hf	0.099 ± 0.003	
Mg	0.0052 ± 0.0003	Mn	0.003 ± 0.002	Мо	0.60 ± 0.01	N (0.00026 ± 0.00004	
Nb	0.0053 ± 0.0007	Ni	60.7 ± 0.3	0	0.0004 ± 0.0001	Р	0.002 ± 0.001	
Re	2.90 ± 0.03	S	0.0003 ± 0.0001	Si	0.028 ± 0.009	Ta	6.5 ± 0.1	
Ti	0.991 ± 0.008	V	0.002 ± 0.001	W	6.45 ± 0.07	Zr	0.005 ± 0.003	

Indicative Values listed in ppm

As (3) Bi	(0.1) Ca	ı (<50) F	Pb (0.2)	Sb (0.2)	Sn (2)	Y	(<20)
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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.

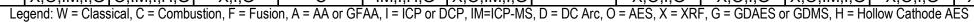
Interpretation of Data

- 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.

	Αl	As	В	Bi	С	Ca	Со	Cr	Cu	Fe	Hf	Mg	Mn	Мо	N	Nb
1	5.734	0.000024	0.0005	0.0000004	0.0029	0.0015	9.215	5.993	0.0009	0.0187	0.0945	0.0043	0.0004	0.5719	0.0002	0.004
2	5.737	0.0002	0.00064	0.00001	0.003	0.0016	9.29	6.057	0.001	0.0192	0.096	0.005	0.0004	0.583	0.0002	0.004
3	5.7591	0.0002	0.0007	0.00001	0.0032	0.0022	9.3306	6.26	0.001	0.0199	0.0961	0.005	0.0007	0.5834	0.0002	0.0041
4	5.763	0.00021	0.0008	0.00001	0.0036	< 0.001	9.362	6.299	0.001	0.0205	0.0972	0.005	0.001	0.584	0.00022	0.0045
5	5.768	0.0003	0.0008	0.00002	0.0037	< 0.0010	9.367	6.3	0.0012	0.022	0.0975	0.0051	0.0011	0.593	0.00023	0.005
6	5.77	0.0008	0.0009	<0.000001	0.0038	<0.005	9.418	6.3091	0.01	0.025	0.0998	0.0052	0.0021	0.5962	0.00024	0.005
7	5.776		0.001	<0.000001	0.00388		9.4561	6.3099	0.0103	0.025	0.1019	0.00527	0.0022	0.598	0.00025	0.0051
8	5.7826		0.001	<0.0001	0.0039		9.48	6.3133	0.0112	0.0253	0.102	0.0056	0.0044	0.599	0.0003	0.0053
9	5.787		0.001		0.004		9.4922	6.318	0.013	0.0278	0.1028	0.0058	0.008	0.599	0.0003	0.0056
10	5.801		0.0011		0.0041		9.5179	6.335	0.022	0.0286	0.106	0.006	0.009	0.6116	0.00032	0.0056
11	5.84		0.0012		0.0042		9.527	6.347		0.0292				0.617	0.0004	0.0057
12	5.847		0.0012		0.00489		9.53	6.381		0.047				0.618		0.007
13	5.85				0.0049		9.55	6.552		0.0503				0.619		0.008
14	5.893						9.55	6.648						0.63		
15							9.676	6.76						0.6357		
Mean	5.79	0.0003	0.0009	0.00001	0.0039	0.0018	9.45	6.3	0.007	0.028	0.099	0.0052	0.003	0.6	0.00026	0.0053
STDV.	0.05	0.0003	0.0002	0.000007	0.0006	0.0004	0.1	0.2	0.007	0.01	0.004	0.0005	0.003	0.02	0.00006	0.001
Certified	5.79	0.0003	0.0009	(0.00001)	0.0039	(<0.005)	9.45	6.3	0.007	0.028	0.099	0.0052	0.003	0.60	0.00026	0.0053
95% C.I.	0.03	0.0003	0.0001		0.0004		0.07	0.1	0.005	0.006	0.003	0.0003	0.002	0.01	0.00004	0.0007
Methods	X,O,I,G		O,IM,I,G	IM,H,G	С	IM,I	X,O,I,G	X,O,I,G	X,O,IM,I,G	X,O,IM,I,G	X,O,IM,I	O,IM,I	X,O,IM,I,G	X,O,I,G	F	X,O,IM,I,G
	Ni	0	Р	Pb	Re	S	Sb	Si	Sn	Та	Ti	V	W	Υ	Zr	
1	59.845	0.0002	0.0003	0.0000026	2.81	0.0002	0.0000044	0.0085	0.00014	6.031	0.97	0.0002	6.165	0.00005	0.0014	
2	60.183	0.0002	0.0003	0.0000020	2.842	0.0002	0.0000044	0.0003	0.00014	6.2809	0.971	0.0002	6.2276	0.00003	0.0014	
3	60.564	0.0002	0.0007	0.00002	2.86	0.0002	0.000012	0.0109	0.0002	6.37	0.971	0.0005	6.4	0.0012	0.0028	
4	60.627	0.0002	0.0007	0.00002	2.87	0.0002	0.00002	0.0103	0.0002	6.384	0.98	0.0003	6.41	<0.0012	0.003	
5	60.67	0.0003	0.0011	<0.00002	2.8865	0.00021	0.00002	0.0117	0.0003	6.4107	0.98	0.000	6.412	<0.0001	0.0031	
6	60.67	0.0003	0.0021	<0.00001	2.908	0.00021	<0.00002	0.0183		6.451	0.9838	0.001	6.418	<0.001	0.0031	
7	60.7379	0.00037	0.0021	<0.00001	2.9086	0.0003	<0.0001	0.0251		6.463	0.99	0.001	6.45	<0.010	0.0032	
8	60.76	0.0004	0.00223	<0.0001	2.91	0.0003		0.0251		6.491	0.994	0.002	6.468	30.010	0.0033	
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•				13.555			[
			2.000				[1	2.000				
	5 5										1.0066					
															J.J.	
	60.7	0.0004	0.002	0.00002	2.9	0.0003	0.000015		0.0002		0.991	0.002		0.001	0.005	
STDV.	0.4	0.0002	0.001	0.000009	0.04	0.0002	0.000007	0.02	0.00007	0.2	0.01	0.002	0.1	0.0007	0.005	
Certified	60.7	0.0004	0.002	(0.00002)	2.90	0.0003	(0.00002)	0.028	(0.0002)	6.5	0.991	0.002	6.45	(<0.002)	0.005	
	0.3	0.0001	0.001	`	0.03	0.0001		0.009		0.1	0.008	0.001	0.07	, , ,	0.003	
95% C.I.													-			
9 10 11 12 13 14 15 Mean	60.8 60.86 61.522	0.0004 0.00048 0.0005 0.0008	0.0031 0.005	<0.005	2.914 2.918 2.938 2.9502 2.97	0.00031 0.0004 0.0006 0.0007		0.031 0.0323 0.0344 0.037 0.0482 0.053 0.065	0.0002	6.492 6.5158 6.53 6.534 6.54 6.561 6.87	0.9943 0.9958 1 1.0066 1.01 1.014	0.0027 0.006	6.4827 6.489 6.4971 6.51 6.53 6.544 6.687	0.001	0.0046 0.005 0.006 0.0212	





Participating Laboratories

Alcoa Howmet, Dover Alloy ATI Specialty Materials, Monroe Timet - Vallejo Dirats Laboratories NSL Analytical Services IMR Test Labs

ATI Powder Metals

Monroe, NC Vallejo, CA Westfield, MA Cleveland, OH Lansing, NY Pittsburgh, PA

Dover, NJ

LECO Corporation
Laboratory Testing, Inc.
Alcoa Howmet, Exeter Alloy
Special Metals IncoTest
ATI Specialty Materials, Lockport
Alcoa Howmet, Research Center

Hatfield, PA
Exeter Devon, England
Hereford, UK
Lockport, NY
Whitehall, MI

St. Joseph, MI

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 AR1653	BCS 462/1	IARM 53D	LECO 501-502	LECO 502-102	NIST 1249	NIST 3114	NIST 3151	TIMET AG1015
ALPHA AR512	BICTA 1	IARM 62A	LECO 501-503	LECO 502-265	NIST 131F	NIST 3126A	NIST 3155	TIMET AR3017
ALPHA AR644	BICTA 2	IARM 68B	LECO 501-505	LECO 502-411	NIST 131G	NIST 3128	NIST 3161A	TIMET B1900
ALPHA AR645	BS 198	IH AC1102	LECO 501-550	LECO 502-414	NIST 1754	NIST 3131A	NIST 3163	TIMET V5100
ALPHA AR892	BS 750A	IH AD1029	LECO 501-643	LECO 502-416	NIST 3101A	NIST 3132	NIST 3165	TRAMPS 2000
ALPHA AR892C	BS CSN-4	IH AD1056	LECO 501-644	LECO 502-456	NIST 3102A	NIST 3133	NIST 3167A	VHG 002-0061R
ALPHA AR946	BS H3B	IH AG1015	LECO 501-645	LECO SS345	NIST 3103A	NIST 3134	NIST 3169	VHG 007-0054R
BAS 346A	HM PWA1484	IH AG1026	LECO 501-646	MBH 212X11233-E	NIST 3106	NIST 3136	NIST 345A	VHG 012-0164F
BCS 345	HM R6-R17	IH AG1087	LECO 501-673	NIST 3162A	NIST 3107	NIST 3137	NIST C2402	VHG 411-0031
BCS 346	IARM 190A	IH N07204	LECO 501-991	NIST 101G	NIST 3109A	NIST 3139A	TELE. PWA 1426	VHG 907-0147
BCS 351	IARM 332A	IH R1780	LECO 501-992	NIST 1191	NIST 3112A	NIST 3143	TELE. RE2-5	
BCS 454/1	IARM 50B	IH V1080	LECO 502-064	NIST 1192	NIST 3113	NIST 3150	TIMET AD1026	

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:

<u>Certifying Body:</u> 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.

<u>Certified Reference Material (CRM):</u> 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.

<u>Inter-Laboratory Analysis Program (ILAP):</u> 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.

<u>Selection of Materials:</u> 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

