

ECISS
EUROPEAN COMMITTEE FOR IRON AND STEEL STANDARDISATION
COMITÉ EUROPÉEN DE NORMALISATION DU FER ET DE L'ACIER
EUROPÄISCHES KOMITEE FÜR EISEN-UND STAHLNORMUNG

EUROPEAN CERTIFIED REFERENCE MATERIAL (EURONORM – CRM)

CERTIFICATE OF CHEMICAL ANALYSIS

EURONORM – CRM No. 691-1, IRON ORE

LABORATORY MEANS (4 values) - Mass content in %

Line No	Fe	Si	Ca	Mg	Al	Ti	Mn	P	S	Na	K	V	Cr	Ni	C
1	64.10	0.5325	---	---	0.4532	0.9295	0.1654	0.0813	0.0581	0.0138	0.0446	0.0552	0.0080	0.0265	---
2	64.12	0.5384	0.9652	1.9448	0.4538	0.9439	0.1677	0.0820	0.0592	0.0139	0.0455	0.0554	0.0083	0.0281	0.2899
3	64.14	0.5385	0.9776	1.9494	0.4583	0.9443	0.1678	0.0831	0.0608	0.0142	0.0473	0.0570	0.0087	0.0283	0.2982
4	64.15	0.5411	0.9840	1.9691	0.4606	0.9469	0.1689	0.0845	0.0608	0.0145	0.0482	0.0584	0.0087	0.0284	0.2988
5	64.16	0.5505	0.9849	1.9989	0.4612	0.9470	0.1699	0.0849	0.0610	0.0147	0.0489	0.0584	0.0090	0.0286	0.3008
6	64.21	0.5514	0.9873	2.0040	0.4636	0.9486	0.1703	0.0873	0.0614	0.0155	0.0497	0.0587	0.0090	0.0288	0.3011
7	64.27	0.5523	0.9894	2.0065	0.4638	0.9523	0.1712	0.0878	0.0643	0.0163	0.0500	0.0589	0.0090	0.0291	0.3020
8	64.27	0.5588	0.9904	2.0067	0.4682	0.9555	0.1712	0.0878	0.0644	0.0165	0.0500	0.0592	0.0092	0.0293	0.3064
9	64.29	0.5614	0.9917	2.0208	0.4722	0.9575	0.1718	0.0882	0.0648	0.0170	0.0506	0.0597	0.0098	0.0295	0.3069
10	64.36	0.5614	0.9917	2.0228	0.4726	0.9624	0.1724	0.0889	0.0650	0.0173	0.0511	0.0597	0.0100	0.0298	0.3097
11	64.37	0.5629	0.9980	---	0.4739	0.9640	0.1724	0.0896	0.0653	0.0189	0.0514	0.0604	0.0101	0.0300	0.3112
12	64.41	0.5634	0.9993	2.0278	0.4776	0.9694	0.1733	0.0900	0.0664	0.0190	0.0516	0.0610	0.0105	0.0302	0.3165
13	64.42	0.5671	0.9995	2.0350	0.4794	0.9785	0.1739	0.0900	0.0666	0.0191	0.0517	0.0623	0.0105	0.0303	0.3252
14	64.45	0.5693	1.0028	2.0397	0.4803	0.9806	0.1750	0.0902	0.0672	0.0192	0.0520	0.0631	0.0110	0.0305	0.3288
15	64.45	0.5700	1.0045	2.0550	0.4836	0.9837	0.1759	0.0904	---	---	0.0525	0.0632	0.0111	0.0314	---
16	64.53	0.5701	1.0095	2.0558	0.4869	0.9859	0.1765	0.0905	---	---	0.0529	0.0641	---	0.0316	---
17	64.64	---	1.0217	2.0592	0.4933	0.9865	0.1779	0.0910	---	---	0.0533	0.0650	---	---	---
18	64.67	---	1.0285	2.0705	0.4960	0.9903	0.1795	0.0917	---	---	0.0561	0.0664	---	0.0334	---
19	64.69	---	1.0289	2.1035	0.4986	0.9918	0.1827	---	---	---	---	---	---	0.0346	---
20	64.69	---	1.0292	---	0.5063	1.0012	0.1837	---	---	---	---	---	---	---	---
21	64.70	---	---	---	---	---	---	---	---	---	---	---	---	---	---
M_M	64.39	0.5556	0.9992	2.0217	0.4752	0.9660	0.1734	0.0877	0.0632	0.0164	0.0504	0.0603	0.0095	0.0299	0.3073
S_M	0.21	0.0125	0.0180	0.0422	0.0154	0.0202	0.0049	0.0033	0.0030	0.0021	0.0029	0.0032	0.0010	0.0020	0.0111
S_w	0.10	0.0063	0.0084	0.0122	0.0070	0.0061	0.0024	0.0016	0.0014	0.0009	0.0012	0.0010	0.0005	0.0005	0.0040

Line No	Zn	Pb	Cu	Fe (II)
1	0.0154	0.0006	---	---
2	0.0158	0.0007	---	20.54
3	0.0164	0.0007	0.0726	20.58
4	0.0165	0.0007	0.0728	20.64
5	0.0168	0.0008	0.0752	20.66
6	0.0173	0.0008	0.0764	20.67
7	0.0180	0.0008	0.0765	20.77
8	0.0181	0.0009	0.0766	20.78
9	---	0.0011	0.0768	20.79
10	0.0185	0.0011	0.0769	20.83
11	0.0190	---	0.0772	20.86
12	0.0198	---	0.0773	---
13	0.0209	---	0.0774	---
14	0.0216	---	0.0780	---
15	0.0216	---	0.0782	---
16	0.0220	---	0.0782	---
17	0.0221	---	0.0794	---
18	0.0222	---	0.0796	---
19	0.0225	---	---	---
20	0.0227	---	---	---
21	0.0233	---	---	---
M_M	0.0195	0.0008	0.0768	20.71
S_M	0.0027	0.0002	0.0020	0.11
S_w	0.0008	0.0001	0.0010	0.05

Line No	Ba	Ce	Sr	Zr	GOI ⁽¹⁾
1	0.0060	0.0030	0.0143	0.0104	---
2	0.0064	0.0030	0.0149	0.0122	1.28
3	0.0065	0.0048	0.0157	0.0123	1.30
4	0.0068	0.0051	0.0160	0.0141	1.31
5	0.0071	0.0059	0.0163	0.0142	1.35
6	0.0072	---	0.0177	0.0145	1.35
7	0.0075	---	0.0178	---	1.44
8	0.0082	---	---	---	1.44
9	---	---	---	---	1.46
10	---	---	---	---	1.47
11	---	---	---	---	1.50
12	---	---	---	---	---
13	---	---	---	---	1.52
14	---	---	---	---	1.55
15	---	---	---	---	1.56

M_M: Mean of intralaboratory means

S_M: Standard deviation of intralaboratory means

S_w: Intralaboratory standard deviation

The laboratory mean values have been examined statistically to eliminate outlying values.

Where a "----" appears in the table it indicates that an outlying value has been eliminated by either the Cochran or Grubbs Test.

Values given *in italic* are for information only.

(1) GOI = Gain On Ignition
(negative Loss On Ignition)

Additional values for information (in µg/g):

As: 0.6; B: 3.2; Be: < 0.1; Cd: < 0.1; Co: 216; Cs: < 0.1; Ga: 21; Hf: 4.7; La: 25; Li: 0.9; Mo: 1.1; Nb: 12; Nd: 32; Rb: 2; Sb: < 0.1; Sc: 46; Sn: 21; Ta: 2; Te: < 0.1; Th: 15; U: 4.2; Y: 5.4

CERTIFIED VALUES - Mass content in %

	Fe	Si	Ca	Mg	Al	Ti	Mn	P	S	Na
M_M	64.39	0.556	0.999	2.022	0.475	0.966	0.1734	0.0877	0.0632	0.0164
C(95 %)	0.10	0.007	0.009	0.022	0.008	0.010	0.0023	0.0017	0.0017	0.0012

	K	V	Cr	Ni	C	Zn	Pb	Cu	Fe (II)
M_M	0.0504	0.0603	0.0095	0.0299	0.307	0.0195	0.0008	0.0768	20.71
C(95 %)	0.0014	0.0016	0.0006	0.0010	0.007	0.0013	0.0002	0.0011	0.08

The half-width confidence interval C(95%) = $\frac{t \times S_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 sections 6.1 and 10.5.2

This certified reference material was prepared in accordance with the recommendations set out in ISO Guides 30 – 35 and issued by:



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On behalf of: The Iron and Steel Nomenclature Co-ordinating Committee (COCOR) of the ECISS, after approval by all the participating laboratories and all the producing organisations (France - ArcelorMittal Maizières Research SA & CTIF; Germany - Iron and Steel CRM Working Group; Stahlinstitut VDEh, Bundesanstalt für Materialforschung und -prüfung (BAM) & MPI für Eisenforschung; Nordic Countries - Nordic CRM Working Group).

METHODS USED

Element	Line number	Methods
Fe	1.2.3.4.5.6.8.9.10.13.14.15.20.21	Titration with Cr (VI) after reduction with Sn (II)
	12 7.11.16.17.18.19	Titration with Cr (VI) after reduction with Ti (III) XRF
Si	1.4.12	ICP-OES
	2	Gravimetry, dehydration with hydrochloric acid
	3.5.8.11.13.15	XRF
	6	FAAS
	7 9 10.16 14	Gravimetry, dehydration with sulphuric acid Gravimetry, dehydration with hydrochloric acid in presence of gelatine Gravimetry, dehydration with perchloric acid ICP-MS
Ca	2	ICP-MS
	3.5.9.10.11.14.15.20	XRF
	4	Complexometric titration, visual end point
	6.7.13.16.18.19 8.12.17	ICP-OES FAAS
Mg	2.5.6.8.13.15.16.17	ICP-OES
	3.4.9.10.12.14.19	XRF
	7.18	FAAS
Al	1	FAAS, without separation
	2.6.11.14.16.17.19.20	XRF
	3.4.5.7.9.10.12.13.15.18	ICP-OES
	8	ICP-MS
Ti	1.2.3.9.10.11.14.15.18	ICP-OES
	4.6.7.8.12.13.16.19.20	XRF
	5	FAAS
	17	ICP-MS
Mn	1.3.4.5.8.10.11.14.17	ICP-OES
	2.7.18.20	FAAS
	6.9.12.15.16.19	XRF
	13	ICP-MS
P	1.2.6.8.10.12.16	XRF
	3.17	ICP-MS
	4.7.9.11.14.15	ICP-OES
	5	Spectrophotometry, molybdenum blue, without extraction
	13.18	Spectrophotometry, phosphovanadomolybdate, extraction
S	1	Combustion, Oxidation reduction titration
	2.4.5.6.7.8.10.12.13.14	Combustion, Infrared absorption
	3	XRF
	9	ICP-MS
	11	ICP-OES
Na	1.2.9.11	ICP-OES
	3.4.5.7.10.13.14	FAAS
	6.8	ICP-MS
	12	XRF
K	1.7.12.13.15.17	FAAS
	2.3.8.18	XRF
	4.9.10.11.14.16	ICP-OES
	5.6	ICP-MS
V	1.13.15	ICP-MS
	2.3.5.6.7.9.11.12.16.18	ICP-OES
	4.10.14.17	XRF
	8	FAAS
Cr	1.3.4.6.9.11.12.13	ICP-OES
	2.10	XRF
	5.7.8.15	ICP-MS
	14	FAAS
Ni	1.2	FAAS
	3.4.5.6.7.8.10.13.15.19	ICP-OES
	9.11.14.16	ICP-MS
	12.18	XRF
C	2.14	Combustion, Gravimetry
	3.4.5.6.7.8.9.10.11.12	Combustion, Infrared absorption
	13	Combustion, Gas volumetry
Zn	1.7.11.20	ICP-MS
	2.3.4.6.12.18.21	ICP-OES
	5.8.10.13.16.19	FAAS
	14.15.17	XRF
Pb	1.2.3.5.6.10	ICP-MS
	4	ETAAS
	7.8.9	ICP-OES
Cu	3.4.7.10.12.15.16.17	ICP-OES
	5.9.11	FAAS
	6.14	XRF
	8.13.18	ICP-MS
Fe (II)	2	Titration with Cr (VI) in the residue of the dissolution in ferric-chloride medium
	3	Titration with Cr (VI) in the residue of the dissolution in bromine-methanol medium
	4.5.7.8.9.11	Titration with Cr (VI) after hydrochloric acid dissolution under CO ₂
	6	Titration with Cr(VI) after sulfuric/hydrofluoric dissolution
	10	Titration with Mn (VII) after hydrochloric acid dissolution under CO ₂
Ba	1.3.6.8	ICP-OES
	2.4.5.7	ICP-MS
	9	XRF
Ce	1.2	ICP-OES
	3.4.5	ICP-MS
Sr	1.2.4.6	ICP-MS
	3.5.7	ICP-OES
Zr	1.2.3.6	ICP-MS
	4.5	ICP-OES
GOI	1	Combustion at 1100°C, Gravimetry
	2	Combustion at 950°C, Gravimetry
	3.4.5.7.8.9.11.12.14.15	Combustion at 1000°C, Gravimetry
	6	Combustion at 1050°C, Gravimetry
	10.13	Combustion at 900°C, Gravimetry

Abbreviations:

ETAAS Electrothermal Atomic Absorption Spectrometry
 FAAS Flame Atomic Absorption Spectrometry
 ICP-MS Inductively Coupled Plasma – Mass Spectrometry

ICP-OES Inductively Coupled Plasma – Optical Emission Spectrometry
 XRF X-Ray Fluorescence

DESCRIPTION OF THE SAMPLE

The sample is available in the form of a powder passing a nominal 100 µm aperture. It is supplied in bottles containing 100 g.

INTENDED USE & STABILITY

ECRM 691-1 is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

It will remain stable provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the content should become discoloured (e.g. oxidised) due to atmospheric contamination it should be discarded.

TRACEABILITY

The traceability of ECRM 691-1 has been established in accordance with principles of ISO Guides 30 – 35 and the International vocabulary of basic and general terms in metrology.

The assigned values for each material are achieved by inter-laboratory characterization, each laboratory using the method of their choice, details of which are given above. These methods are either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds. Most methods used were either international or national standard methods or methods which are technically equivalent.

PARTICIPATING LABORATORIES

AG der Dillinger Hüttenwerke, Dillingen/Saar (Germany)
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ArcelorMittal Asturias, Avilés (Spain)
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