

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. 269-1, HIGH ALLOY STEEL

LABORATORY MEANS (4 values) - Mass content in %

Line No	C	Si	Mn	P	S	Cr	Mo	Ni	As	Co	Cu	N	Nb
1	0.0473	---	1.2285	0.0295	0.0006	---	0.3806	---	0.0056	---	0.3505	0.0421	0.0211
2	0.0486	0.4270	---	0.0295	0.0007	---	0.3868	7.975	0.0056	---	0.3563	0.0447	0.0220
3	0.0489	0.4279	1.2456	0.0296	0.0008	18.048	0.3871	7.988	0.0056	0.1072	0.3563	0.0449	0.0227
4	0.0490	0.4293	1.2466	0.0298	0.0008	18.080	0.3882	7.996	0.0058	0.1079	0.3597	0.0451	0.0236
5	0.0490	0.4317	1.2475	0.0303	0.0009	18.091	0.3889	8.003	0.0058	0.1088	0.3614	0.0451	0.0236
6	0.0493	0.4346	1.2520	0.0305	0.0009	18.105	0.3934	8.025	0.0060	0.1094	0.3624	0.0451	0.0239
7	0.0497	0.4348	1.2542	0.0308	0.0010	18.128	0.3942	8.035	0.0060	0.1103	0.3630	0.0451	0.0240
8	0.0498	0.4353	1.2563	0.0309	0.0011	18.128	0.3946	8.037	0.0061	0.1112	0.3634	0.0459	0.0241
9	0.0498	0.4386	1.2578	0.0311	0.0011	18.130	0.3962	8.037	0.0061	0.1119	0.3642	0.0460	0.0241
10	0.0499	0.4387	1.2605	0.0313	0.0011	18.133	0.3963	8.043	0.0062	0.1121	0.3642	0.0466	0.0242
11	0.0501	0.4418	1.2617	0.0313	0.0012	18.149	0.3967	8.050	0.0062	0.1124	0.3666	0.0471	0.0245
12	0.0517	0.4420	1.2627	0.0315	0.0013	18.150	0.3970	8.054	0.0063	0.1124	0.3669	0.0479	---
13	0.0526	0.4446	1.2640	0.0315	0.0014	18.159	0.3970	8.056	0.0064	0.1126	0.3680	0.0482	0.0250
14	0.0527	0.4450	1.2650	0.0315	0.0014	18.195	0.3991	8.063	0.0064	0.1129	0.3683	0.0497	0.0254
15	---	0.4456	1.2650	0.0316	---	18.202	0.4021	8.064	0.0064	0.1129	0.3694	---	0.0257
16	---	0.4462	1.2785	0.0321	---	18.203	0.4033	8.068	0.0068	0.1130	0.3698	---	0.0258
17	---	0.4464	1.2800	0.0324	---	18.211	0.4049	8.073	---	0.1133	0.3698	---	0.0274
18	---	0.4486	1.2913	0.0326	---	18.212	0.4060	8.100	---	0.1134	0.3715	---	---
19	---	0.4498	1.2946	0.0336	---	18.219	0.4082	8.131	---	0.1156	0.3730	---	---
20	---	0.4505	---	0.0343	---	---	0.4083	---	---	---	0.3742	---	---
21	---	0.4573	---	---	---	---	0.4103	---	---	---	0.3759	---	---
M_M	0.0499	0.4408	1.2618	0.0313	0.0010	18.150	0.3971	8.044	0.0061	0.1116	0.3655	0.0460	0.0242
S_M	0.0016	0.0084	0.0165	0.0013	0.0003	0.052	0.0081	0.039	0.0004	0.0023	0.0064	0.0019	0.0016
S_w	0.0006	0.0038	0.0079	0.0007	0.0001	0.061	0.0040	0.044	0.0003	0.0015	0.0035	0.0004	0.0005

Line No	Sn	Ti	V	W
1	0.0085	0.0004	0.0962	0.0278
2	0.0087	0.0004	0.0972	0.0283
3	0.0091	0.0004	0.0975	0.0293
4	0.0093	0.0005	0.0976	0.0294
5	0.0094	0.0005	0.0984	0.0298
6	0.0097	0.0005	0.0987	0.0299
7	0.0098	0.0007	0.0988	0.0303
8	0.0100	0.0008	0.0989	0.0303
9	0.0102	0.0008	0.0990	0.0304
10	0.0102	0.0009	0.0992	0.0308
11	0.0103	---	0.0993	0.0312
12	0.0103	---	0.0995	0.0312
13	0.0103	---	0.0996	0.0320
14	0.0103	---	0.0996	0.0329
15	0.0108	---	0.1001	0.0331
16	0.0110	---	0.1001	0.0336
17	---	---	0.1003	---
18	---	---	0.1013	---
19	---	---	0.1019	---
20	---	---	---	---
21	---	---	---	---
M_M	0.0099	0.0006	0.0991	0.0306
S_M	0.0008	0.0002	0.0014	0.0017
S_w	0.0003	0.0001	0.0014	0.0006

Al(tot)	Pb
0.0006	< 0.0001
0.0008	< 0.0001
0.0008	< 0.0001
0.0015	< 0.0001
0.0019	< 0.0001
< 0.0020	0.0001
0.0024	0.0001
0.0028	0.0001
0.0029	0.0001
0.0032	0.0001
0.0035	0.0005
0.0036	0.0008
---	0.0011
---	---
---	< 0.0020
---	< 0.0020
---	---

M_M: Mean of the intralaboratory means
S_M: Standard deviation of the 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 reported as "less than" by the participating laboratories have not been taken into account in the statistical calculations.

Values given *in italic* are for information only.

Additional values for information (in µg/g):

B: < 1; Bi: < 0.02; Ce: 0.06; Ga: 24; La: 0.02; Mg: < 5; Sb: 12; Se: < 2; Ta: < 1; Te: < 0.01; Zn: 4; Zr: < 1

CERTIFIED VALUES - Mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	As
M_M	0.0499	0.441	1.262	0.0313	0.0010	18.150	0.397	8.044	0.0061
C(95 %)	0.0009	0.004	0.009	0.0007	0.0002	0.027	0.004	0.020	0.0002

	Co	Cu	N	Nb	Sn	Ti	V	W
M_M	0.1116	0.366	0.0460	0.0242	0.0099	0.0006	0.0991	0.0306
C(95 %)	0.0012	0.003	0.0011	0.0009	0.0004	0.0002	0.0007	0.0009

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, BAM Bundesanstalt für Materialforschung und -prüfung & MPI für Eisenforschung; Nordic Countries - Nordic CRM Working Group).

METHODS USED

Element	Line number	Methods
C	1.2.3.4.5.6.7.9.10.11.12.13.14	Combustion, infrared absorption
	8	Combustion, non-aqueous titration after absorption in organic solvent
Si	2.3.8.13.17	Gravimetry, dehydration with perchloric acid
	4.5.6.7.9.10.11.12.15.21	ICP-OES
	14.18	MAS, molybdenum blue, without extraction
	16	Gravimetry, dehydration with hydrochloric acid
	19	ICP-MS
Mn	20	FAAS
	1.4.7.8.9.10.11.12.13.15.16.17	ICP-OES
	3.5.6.14.18	MAS, periodate oxidation
P	19	FAAS
	1.6.8	MAS, molybdenum blue, without extraction
	2.10.15.17.20	MAS, phosphovanadomolybdate, extraction
	3.14	ICP-MS
	4	MAS, molybdenum blue, extraction
S	5.7.9.11.12.13.16.18.19	ICP-OES
	1.2.3.4.5.6.7.8.9.10.11.14	Combustion, infrared absorption
	12	MAS, methylene blue, evolution as H ₂ S in hypophosphoric and formic acid medium
	13	Gravimetry as BaSO ₄ without separation
	3.4.6.10.11.12.15.16.17.18	ICP-OES
Cr	5.7.8.9	Titration with Fe (II), oxidation with persulphate
	13.14	Titration with Fe (II), oxidation with peroxide
	19	Titration with Fe (II), oxidation with perchloric acid
	1.2.4.5.6.8.9.10.11.12.14.15.17.18.19.21	ICP-OES
Mo	3.13.20	MAS, thiocyanate in presence of Sn (II), extraction
	7	MAS, thiocyanate in presence of ascorbic acid, extraction
	16	ICP-MS
	2	Titration with dichromate, separation with dimethylglyoxime
Ni	3.9.13	Gravimetry, dimethylglyoxime
	4.6.7.8.11.12.14.15.16.17.18.19	ICP-OES
	5	Cyanometric titration
	10	MAS, dimethylglyoxime, extraction
	1	MAS, DDC, separation as arsine
As	2.6.11.12.15	ETAAS
	3	AAS, evolution as arsine
	4.7.10.13.14	ICP-OES
	5	ICP-OES, evolution as arsine
	8.9.17	ICP-MS
	16	Titration with iodine, precipitation of elemental As
Co	3.4.5.6.7.8.9.10.11.12.14.15.16.17.18	ICP-OES
	13	ETAAS
	19	ICP-MS
Cu	1.4	FAAS
	2.3.5.6.8.9.10.11.12.13.15.16.17.18.19.20.21	ICP-OES
	7	ICP-MS
	14	MAS, DDC, extraction
N	1.2.3.4.5.6.7.8.10.11.12	Thermal conductivity, decomposition in graphite crucible
	9.13.14	Acidimetric titration after distillation, visual end point
Nb	1.7	ICP-MS
	2.3.4.5.6.8.9.10.11.13.14.15.16.17	ICP-OES
Sn	1.3.5.7.8.9.10.11.12.15	ICP-OES
	2.4.16	ETAAS
	6.13	ICP-MS
	14	FAAS, extraction with TOPO/KI/MIBK
Ti	1.7.9	ICP-MS
	2.3.4.5.6.8.10	ICP-OES
V	1.2.3.4.6.7.8.9.10.11.12.14.15.16.17.18.19	ICP-OES
	5	ICP-MS
	13	MAS, hydrogen peroxide
W	1.2.4.5.6.7.8.11.12.13.14.15.16	ICP-OES
	3.9.10	ICP-MS
<i>Al(tot)</i>	1.3.5.7.8.9.11.12	ICP-OES
	2.6	ICP-MS
	4	ETAAS
	10	FAAS, without separation
<i>Pb</i>	1.2.4.5.7.9.11	ETAAS
	3.8.10	ICP-MS
	6.16	ICP-OES
	12.17	FAAS
	13	MAS, dithizone

Abbreviations:

AAS Atomic Absorption Spectrometry
ETAAS Electrothermal Atomic Absorption Spectrometry
DDC Diethyldithiocarbamate
FAAS Flame Atomic Absorption Spectrometry
ICP-OES Inductively Coupled Plasma – Optical Emission Spectrometry

ICP-MS Inductively Coupled Plasma – Mass Spectrometry
MAS Molecular Absorption Spectrometry
MIBK Methylisobutylketone (4-methyl pentan-2-one)
TOPO Tri-octylphosphine oxide

DESCRIPTION OF THE SAMPLE

The sample consists of chips passing a nominal 1000 µm aperture sieve from which the fines passing a nominal 250 µm sieve has been removed. It is supplied in bottles containing 100 g [ref ECRM 269-1(C)].
It is also supplied in the form of 35 mm dia. x 25 mm discs [ref ECRM 269-1 (D)].

INTENDED USE & STABILITY

ECRM 269-1(C) 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.

The solid (disc) sample, ECRM 269-1(D), is intended for establishing and checking the calibration of optical emission and X-ray spectrometers for the analysis of samples of similar materials. The “as received” working surface of the sample should be finished before use to remove any protective coating. It will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

TRACEABILITY

The traceability of ECRM 269-1 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

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FURTHER INFORMATION

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