

ECISS
EUROPEAN COMMITTEE FOR IRON AND STEEL STANDARDIZATION
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. 781-1 SILICON CARBIDE REFRACTORY

LABORATORY MEANS (4 Values)
 mass content in % related to the dried (105°C) sample

Line No.	Total C	Total Si	Total Al
1	—	34.99	4.144
2	47.73	35.08	4.282
3	48.10	35.32	4.303
4	48.11	35.37	4.307
5	48.16	35.39	4.322
6	48.20	35.40	4.340
7	48.23	35.44	4.343
8	48.24	35.50	4.380
9	48.29	35.58	4.400
10	48.31	35.71	4.405
11	48.38	35.74	4.440
12	48.39	35.75	4.445
13	48.40	35.77	4.446
14	48.43	35.81	4.446
15	48.47	35.86	4.454
16		35.86	4.458
17		35.89	4.460
18			4.477
19			4.543
M_M	48.25	35.56	4.389
s_M	0.19	0.28	0.092
s_w	0.26	0.13	0.043

M_M: Mean of the intralaboratory means s_M: Standard deviation of the intralaboratory means
 s_w: Intralaboratory standard deviation s_b: Interlaboratory standard deviation $s_M = \sqrt{s_b^2 + s_w^2/4}$

The laboratory mean values have been examined statistically to eliminate outstanding values. Where a "—" appears in the table it indicates that an outlying value has been omitted by either the Cochran or Grubbs Test.

CERTIFIED VALUES
 mass content in %

	Total C	Total Si	Total Al
M_M	48.25	35.56	4.39
C(95%)	0.11	0.15	0.04

The half-width confidence interval C(95%) = $t \times \frac{s_M}{\sqrt{n}}$ 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 35:1989 section 4.



This reference material was prepared and issued by:

BUREAU OF ANALYSED SAMPLES LIMITED

Newham Hall, Middlesbrough, England

MARCH 1993

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 organizations. (France-IRSID/CTIF Germany-Iron and Steel CRM Working Group, UK-BAS Ltd.)

APPROXIMATE VALUES FOR INFORMATION

Laboratory Means (4 values)
mass content in % related to the dried (105°C) sample

Line No.	C Free	Si Free	Fe Total	Mn	P	Cr	Mo	Ni
1	36.69	4.285	—	—	0.0087	0.0205	—	0.0180
2	36.72	4.347	0.7350	0.0234	0.0095	0.0210	0.0198	0.0183
3	36.95	4.575	0.7361	0.0242	0.0097	0.0222	0.0205	0.0190
4	36.97	4.576	0.7600	0.0262	0.0109	0.0224	0.0241	0.0197
5	37.00	4.592	0.7900	0.0264	0.0112	—	0.0255	0.0200
6	37.09	4.600	0.7918	0.0268	0.0116	0.0227	0.0260	0.0201
7	37.20	4.655	0.7940	0.0272	0.0120	0.0230	0.0271	0.0214
8	37.22	4.665	0.8048	0.0274	0.0122	0.0233	0.0280	—
9	37.23	4.700	0.8057	0.0274	0.0122	0.0235	0.0282	0.0221
10	37.28	4.756	0.8137	0.0274	0.0122	0.0239	0.0294	0.0231
11	37.29	4.778	0.8150	0.0277	0.0125	0.0240	0.0303	0.0236
12	37.32	4.828	0.8180	0.0281	—	0.0244	0.0311	0.0251
13	—	4.835	0.8191	—	0.0136	0.0245	—	—
14	37.57	4.854	0.8325	0.0285	0.0137	0.0254	—	—
15	37.91	—	0.8386	0.0287	0.0140	0.0258	—	—
16	37.93	—	0.8535	0.0288	—	0.0288	—	—
17	—	—	0.8903	0.0292	—	0.0290	—	—
18	—	—	—	0.0306	—	—	—	—
M_M	37.22	4.646	0.8061	0.0274	0.0117	0.0240	0.0264	0.0210

Line No.	B	N	Ti	V	Ca	Mg	Na	K
1	0.0132	0.0148	0.0228	—	0.0364	0.0310	0.0250	0.3499
2	0.0137	0.0165	0.0278	0.0189	0.0365	—	0.0262	0.3520
3	0.0144	0.0260	0.0282	0.0192	0.0375	0.0339	0.0275	0.3528
4	0.0145	0.0300	0.0290	0.0201	0.0388	0.0350	0.0275	0.3561
5	0.0147	0.0330	0.0298	0.0206	0.0405	0.0394	0.0282	0.3595
6	0.0148	0.0335	0.0298	0.0209	0.0416	0.0405	0.0292	0.3650
7	0.0151	0.0366	0.0303	0.0212	0.0418	0.0414	0.0295	0.3702
8	—	0.0367	0.0303	0.0212	0.0428	0.0416	0.0300	0.3705
9	0.0186	—	0.0305	0.0213	0.0435	0.0419	0.0308	0.3724
10	—	—	0.0307	0.0228	0.0438	0.0425	0.0315	0.3730
11	—	—	0.0312	0.0231	0.0455	0.0428	0.0320	0.3792
12	—	—	0.0314	0.0231	0.0482	0.0429	0.0330	0.3818
13	—	—	0.0324	0.0238	0.0485	0.0434	0.0332	0.3834
14	—	—	0.0325	0.0245	0.0486	0.0447	—	0.3863
15	—	—	0.0326	—	0.0495	0.0448	0.0342	0.3893
16	—	—	—	—	0.0498	0.0462	0.0348	0.3900
17	—	—	0.0344	—	—	0.0475	0.0365	0.3908
18	—	—	0.0375	—	—	0.0484	—	0.3914
19	—	—	0.0420	—	—	0.0497	—	0.3975
20	—	—	0.0426	—	—	—	—	0.4185
M_M	0.0149	0.0282	0.0320	0.0216	0.0433	0.0421	0.0308	0.3765

Approximate values (from at least two laboratories) for information only: - O = 10.5%; Fe° = 0.35%

DESCRIPTION OF THE SAMPLE

This sample consists of specially prepared material passing a 125µm. aperture sieve and then passed over a magnetic separator.
It is only supplied in bottles containing 100g.

PARTICIPATING LABORATORIES

Böhler GmbH, Kapfenberg (Austria)
 British Ceramic Research Ltd., Stoke-on-Trent (U.K.)
 British Gas plc, Solihull (U.K.)
 Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin
 (Germany)
 Centro Sviluppo Materiali (CSM), Rome (Italy)
 Cockerill-Sambre SA, Couillet (Belgium)
 Dyson Group, Research and Development Laboratories, Sheffield
 (U.K.)
 Elektroschmelzwerk Kempton GmbH., Gefrath (Germany)
 ENEA, Rome (Italy)
 Feuerfest-Prüflabor FPL GmbH., Bonn (Germany)
 Hepworth Refractories Ltd., Worksop (U.K.)

Hoogovens Groep BV, IJmuiden (Netherlands)
 Huttenwerke Krupp Mannesmann GmbH.
 Institut de Recherches de la Sidérurgie Française (IRSID),
 Maizières-lès-Metz (France)
 Laborlux SA, Esch sur Alzette (Luxembourg)
 Morgan Material Technology Ltd., Stourport-on-Severn (U.K.)
 Rautaruukki Oy, Raahelä (Finland)
 Ridsdale & Co. Ltd., Middlesbrough (U.K.)
 Sollac, Dunkerque (France)
 Sollac, Fos-sur-Mer (France)
 Staatliches Material Prüfungsamt, Nordrhein-Westfalen (MPA-NRW),
 Dortmund (Germany)
 Thyssen Stahl AG, Duisburg-Hamborn (Germany)

METHODS USED
EURONORM – CRM No. 781-1

Element	Line Number	Methods
C Total	2-4-9-10-13-14 3 5-7-8 6-12-15 11	Combustion, coulometric titration Combustion, conductimetry Combustion, gravimetry Combustion, infrared absorption Combustion, non-aqueous titration
Si Total	1-2-4-6-7-8-9-11-12-14 3-5-10-13-16-17 15	Gravimetric, dehydration with perchloric acid XRF Atomic Absorption Spectrometry
Al Total	1-4-5-7-8-11-14-18-19 2-3-6-9 10-12-13-15-16-17	Atomic Absorption Spectrometry ICP-AES XRF
C free	1-3 2-5-8-14-16 4 6 7-9-10 11 12 15	Loss on ignition at 800°C Coulometry after combustion at 800°C Infrared absorption after combustion at 750°C Loss on ignition at 750°C after oxidation at 500°C Difference between total carbon and carbon in ignited sample Gravimetric, ignition at 750°C Non-aqueous titration, ignition at 800°C Conductivity, ignition at 750°C
Si free	1-7-12-13 2 2-3-4-5-8-9-10-11-14 6	Titrimetric, displacement of silver Photometric as silicomolybdate Gas volumetric ICP-AES
Fe total	2 3-11 4-5-6-12-16-17 8-9-10-13-15 14	Photometric, 1-10 phenanthroline XRF ICP-AES Atomic Absorption Spectrometry Photometric, 2,2'-dipyridyl
Mn	2-3-6 5-7-9-15-18 4-8-10-11-12-14-16-17	XRF Atomic Absorption Spectrometry ICP-AES
P	1-5-6 2-4-8-10-14-15 3-9-13 7 11	XRF Photometric, Phosphovanadomolybdate, with extraction ICP-AES Acidimetric titration of ammonium phosphomolybdate Photometric, molybdenum blue, no extraction
Cr	1-10 2-3-7-12-14-17-18 4-6-8-9-11-13-15-16	XRF Atomic Absorption Spectrometry ICP-AES
Mo	2-4-5-10-11 3-6-8-9-12 7	ICP-AES Atomic Absorption Spectrometry XRF
Ni	1-5-6-12 2-3-4-7-9-10-11	ICP-AES Atomic Absorption Spectrometry
B	1-5-9 2-4-6-7 3	Photometric with curcumin ICP-AES Photometric with 1-1 dianthrime after separation
N	1-3-6-7-8 2 4-5	Thermal conductivity, decomposition in graphite crucible Photometric with Nessler reagent after distillation Acidimetric titration after distillation
Ti	1-8-20 2-3-5-6-9-12-13-14-18 4-7-11-15-17 10 19	XRF ICP-AES Atomic Absorption Spectrometry Photometric with diantipyrylmethane Photometric with hydrogen peroxide
V	2-5-7-8-11 3-4-6-9-10-13-14 12	ICP-AES Atomic Absorption Spectrometry XRF
Ca	1-2-4-5-6-12-14 3-10-13-15 7-8-9-11-16	Atomic Absorption Spectrometry XRF ICP-AES
Mg	1-14-18 3-5-6-7-8-11-12-13-15 4-9-10-16-17-19	XRF Atomic Absorption Spectrometry ICP-AES
Na	1-4-9-15 2-3-5-6-7-8-10-11-13-16-17 12	Atomic Emission Spectrometry Atomic Absorption Spectrometry XRF
K	1-2-4-5-7-8-9-10-11-12-15-19 3-14-17-18-20 6 13-16	Atomic Absorption Spectrometry XRF ICP-AES Atomic Emission Spectrometry

Abbreviations: ICP-AES : Inductively Coupled Plasma - Atomic Emission Spectrometry
XRF : X-Ray Fluorescence Spectrometry

FURTHER INFORMATION

For information regarding the preparation, certification and supply of these European Certified Reference Materials (EURONORM-CRMs) and the use of the statistical information given on this certificate, please refer to Information Circulars No. 1 (ECISS) and No. 5 (ECSC), both of which are available from the national standards body in your country. (In the UK this is the BSI, 2 Park Street, London, W1A 2BS).

Des informations complémentaires sur la fabrication, la certification et la distribution des Matériaux de Référence Certifiés Européens (EURONORM-MRC) ainsi que sur l'utilisation des informations statistiques données sur le certificat se trouvent dans les circulaires d'information No. 1 (ECISS) et No. 5 (CECA). On peut se procurer ces deux circulaires auprès des organismes nationaux de normalisation. (Pour la France: AFNOR, Tour Europe - Cedex 7, 92080 Paris La Défense).

Angaben über Herstellung, Zertifizierung und Bezugsmöglichkeiten dieser Zertifizierten Europäischen Referenzmaterialien (EURONORM-ZRM) sowie über die Anwendung der in diesem Zertifikat enthaltenen statistischen Daten finden sich in den Mitteilungen Nr. 1 (ECISS) und Nr. 5 (EGKS), beide zu beziehen durch die nationalen Normenorganisationen. (In Deutschland bei der Vertriebsstelle des DIN: Beuth-Verlag GmbH, Burggrafenstrasse 4-10, 1000 Berlin 30).

Note regarding the presence of metallic iron (Fe^0)

As this sample contains 0.35% metallic iron special care should be taken when making determinations involving fusion of the sample. The presence of metallic iron may also cause problems in the determination of:-

free silicon by silver displacement,
loss on ignition,
free carbon,

unless appropriate precautions are taken or corrections applied.

- Comme cet échantillon contient 0.35% de fer métallique, des précautions particulières doivent être prises au cours des procédures incluant une fusion de l'échantillon. La présence de fer métallique peut également poser des problèmes lors de la détermination:

du silicium libre par déplacement de l'argent,
de la perte au feu,
du carbone libre,

si des précautions ne sont pas prises et si des corrections appropriées ne sont pas appliquées.

- Da diese Probe 0.35% metallisches Eisen enthält, sind besondere Vorsichtsmaßnahmen zu ergreifen, wenn Bestimmungen durchgeführt werden, die ein Aufschmelzen der Probe einschließen.

Die Gegenwart metallischen Eisens kann darüber hinaus Störungen hervorrufen bei der Bestimmung

des freien Siliciums durch Freisetzen von Silber aus Silberfluorid,
des Glühverlustes oder
des freien Kohlenstoffs,

wenn nicht geeignete Vorkehrungen getroffen oder Korrekturen angebracht werden

Note regarding calculated SiO_2 content

$\text{SiO}_2 = 11.7$ (calculated from the total oxygen content and the oxygen present in oxides other than SiO_2)
 ≈ 11.8 (calculated from the total silicon content and silicon present as silicon carbide and free silicon.)

Taking all the elements cited in this certificate into consideration gives a grand total $\approx 100.2\%$