

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. 090-1 1% CARBON STEEL

LABORATORY MEANS (4 Values)
 mass content in %

| Line No. | C | Si | Mn | P | S | Cr | Mo | Ni | N | V |
|----------|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| 1 | - | - | 0.2178 | 0.0116 | 0.0083 | 0.1076 | 0.0078 | 0.0482 | - | - |
| 2 | 1.042 | 0.2650 | 0.2218 | 0.0119 | 0.0085 | 0.1105 | 0.0080 | 0.0504 | 0.0141 | 0.1952 |
| 3 | 1.043 | 0.2728 | 0.2226 | 0.0120 | 0.0088 | 0.1128 | 0.0082 | 0.0504 | 0.0142 | 0.1954 |
| 4 | 1.045 | 0.2743 | 0.2234 | 0.0121 | 0.0089 | 0.1150 | 0.0082 | 0.0506 | 0.0143 | 0.1966 |
| 5 | 1.046 | 0.2744 | 0.2238 | 0.0121 | 0.0090 | 0.1185 | 0.0082 | 0.0508 | 0.0144 | 0.1968 |
| 6 | 1.049 | 0.2768 | 0.2238 | 0.0122 | 0.0090 | 0.1190 | 0.0082 | 0.0509 | 0.0144 | 0.1999 |
| 7 | 1.050 | 0.2778 | 0.2242 | 0.0122 | 0.0091 | 0.1193 | 0.0084 | 0.0510 | 0.0145 | 0.2005 |
| 8 | 1.051 | 0.2786 | 0.2250 | 0.0124 | 0.0092 | 0.1200 | 0.0084 | 0.0514 | 0.0146 | 0.2009 |
| 9 | 1.052 | 0.2798 | 0.2250 | 0.0125 | 0.0094 | 0.1204 | 0.0085 | 0.0517 | 0.0146 | 0.2015 |
| 10 | 1.052 | 0.2806 | 0.2250 | 0.0127 | 0.0094 | 0.1208 | 0.0086 | 0.0520 | 0.0147 | 0.2018 |
| 11 | 1.054 | 0.2835 | 0.2255 | 0.0129 | 0.0094 | 0.1212 | 0.0087 | 0.0527 | 0.0147 | 0.2032 |
| 12 | 1.055 | 0.2836 | 0.2255 | 0.0130 | 0.0095 | 0.1229 | 0.0088 | 0.0527 | 0.0147 | 0.2038 |
| 13 | 1.055 | 0.2850 | 0.2258 | 0.0133 | 0.0095 | 0.1230 | 0.0090 | 0.0529 | 0.0148 | 0.2040 |
| 14 | 1.058 | 0.2850 | 0.2268 | 0.0135 | 0.0100 | 0.1237 | 0.0091 | 0.0536 | 0.0148 | 0.2056 |
| 15 | 1.058 | 0.2857 | 0.2274 | 0.0135 | 0.0100 | 0.1256 | 0.0092 | 0.0544 | 0.0149 | 0.2062 |
| 16 | 1.064 | 0.2860 | 0.2280 | 0.0135 | 0.0100 | 0.1259 | 0.0093 | 0.0545 | 0.0150 | 0.2084 |
| 17 | 1.064 | 0.2865 | 0.2287 | 0.0135 | 0.0100 | 0.1265 | 0.0096 | 0.0549 | 0.0152 | 0.2090 |
| 18 | 1.065 | 0.2895 | 0.2289 | 0.0136 | 0.0104 | 0.1268 | 0.0104 | 0.0572 | | 0.2106 |
| 19 | 1.066 | 0.2898 | 0.2298 | 0.0137 | 0.0107 | 0.1280 | 0.0106 | 0.0588 | | 0.2122 |
| 20 | | 0.2908 | 0.2335 | 0.0141 | 0.0108 | 0.1288 | 0.0110 | 0.0590 | | 0.2184 |
| M_M | 1.054 | 0.2813 | 0.2256 | 0.0128 | 0.0095 | 0.1208 | 0.0089 | 0.0529 | 0.0146 | 0.2037 |
| S_M | 0.008 | 0.0067 | 0.0033 | 0.0007 | 0.0007 | 0.0058 | 0.0009 | 0.0029 | 0.0003 | 0.0061 |
| S_w | 0.003 | 0.0041 | 0.0022 | 0.0005 | 0.0004 | 0.0027 | 0.0005 | 0.0007 | 0.0003 | 0.0024 |

M_M : Mean of the laboratory means S_M : Standard deviation of the laboratory 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 outlying 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 %

| | C | Si | Mn | P | S | Cr | Mo | Ni | N | V |
|--------|-------|-------|-------|--------|--------|-------|--------|-------|--------|-------|
| M_M | 1.054 | 0.281 | 0.226 | 0.0128 | 0.0095 | 0.121 | 0.0089 | 0.053 | 0.0146 | 0.204 |
| C(95%) | 0.005 | 0.003 | 0.002 | 0.0003 | 0.0003 | 0.003 | 0.0004 | 0.002 | 0.0002 | 0.003 |

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 35:1989 section 4.

DESCRIPTION OF THE SAMPLE

This sample is available in the form of chips passing a 1700µm aperture sieve from which the fines passing a 250µm aperture sieve have been removed. It is supplied in bottles containing 100g, ECRM 090-1(C). It is also supplied in the form of 38mm dia discs, ECRM 090-1(D).



This reference material was prepared and issued by:
BUREAU OF ANALYSED SAMPLES LIMITED

Newham Hall, Middlesbrough, England

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: VDEh, BAM & MPI für Eisenforschung, UK-BAS Ltd.)



Certificate No. Q3993

Revised MAY 2000

(First issued in May 1983 before certification of Nitrogen in 1998)

PARTICIPATING LABORATORIES

| | |
|--|---|
| AG der Dillinger Huttenwerke, Dillingen-Saar (Germany) | Hüttenwerke Krupp-Mannesmann GmbH, Duisburg (Germany) |
| Arbed, Division d'Esch-Belval, Esch-sur-Alzette (Luxembourg) | Imphy SA, Imphy (France) |
| Aubert & Duval, Les Ancizes (France) | Inco Test Ltd, Hereford (UK) |
| Automobiles Peugeot, Voujeaucourt (France) | Krupp-Hoesch Stahl AG, Dortmund (Germany) |
| Babcock Power Ltd., Renfrew (UK) | Laboratoire Boudet et Dussaix, Croissy-sur-Seine (France) |
| Böhler Edelstahl GmbH, Kapfenberg (Austria) | Laboratoire National d'Essais, Paris (France) |
| Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany) | Ridsdale & Co. Ltd., Middlesbrough (UK) |
| Centro Technique des Industries de la Fonderie (CTIF), Sèvres (France) | Sanderson Kayser Ltd., Sheffield (UK) |
| Centro Sperimentale Metalurgico (CSM), Rome (Italy) | Soc AFL Falck, Milan (Italy) |
| Cockerill-Sambre, Couillet (Belgium) | SOLLAC, Florange (France) |
| Cockerill-Sambre, Seraing (Belgium) | Stahlwerke Peine-Salzgitter AG, Salzgitter (Germany) |
| Corus (formerly British Steel), Scunthorpe (UK) | Stahlwerke Röchling-Burbach GmbH, Volklingen-Saar (Germany) |
| Daetst, Copenhagen (Denmark) | USINOR, Longwy (France) |
| EWK Edelstahl Witten-Krefeld GmbH, Witten (Germany) | Voest-Alpine Stahl Linz GmbH, Linz (Austria) |
| Hoogovens Staal BV, IJmuiden (Netherlands) | Willan Metals Ltd., Rotherham (UK) |
| Howmet Ltd., Exeter (UK) | |

INTENDED USE & STABILITY

The chip sample, ECRM 090-1(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 stoichiometric metals or 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 contents should become discoloured (eg oxidised) due to atmospheric contamination they should be discarded.

The solid (disc) sample, ECRM 090-1(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 lished before use to remove any protective coating. It will remain stable provided that it is not subjected to excessive heat (eg, during preparation of the working surface).

TRACEABILITY

The traceability of this ECRM is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

METHODS USED EURONORM – CRM No. 090-1

| Element | Line Number | Methods |
|---------|-------------------------------------|--|
| C | 2-4-6-8-10-13-16-17-18-19 | 3 Combustion, infra-red absorption |
| | | 5-7-15 Combustion, conductimetric |
| | | 9-12 Combustion, non-aqueous titration |
| | | 11 Combustion, coulometric titration |
| | | 14 Combustion, thermal conductivity |
| | | 14 Combustion, gravimetric |
| Si | 2-3-4-5-6-8-13-14-15-19-20 | 7-10-12-17 Gravimetric, dehydration with perchloric acid |
| | | 9 FAAS |
| | | 11 ICP AES |
| Mn | 1-3-4-5-6-9-10-12-20 | 11-16-18 Photometric as molybdenum blue |
| | | 2-7-11-13-15-18 Photometric, periodate oxidation |
| | | 8-14-16-19 Photometric, persulphate oxidation |
| P | 1-3-4-5-7-9-10-11-16-19-20 | 17 ICP AES |
| | | 2-6-8-12-13-14-15-17-18 Photometric as molybdenum blue, without extraction |
| | | 1-18 Photometric as phosphovanadomolybdate, after extraction |
| S | 2-3-4-12 | 5 Combustion, oxidation/reduction titration |
| | | 6-7-8-9-10-13-14-16-19-20 Combustion, acidimetric titration, absorption in hydrogen peroxide or silver nitrate |
| | | 5 Combustion, coulometric |
| | | 11 Combustion, infra-red absorption |
| | | 15 Gravimetric as barium sulphate after separation on alumina |
| Cr | 1-2-7-11-19 | 17 Combustion, conductimetric |
| | | 3-4-5-6-10-14-15-16-17-18-20 Combustion, photometric with p-rosaniline |
| | | 8-12 Photometric with diphenylcarbazide |
| | | 9 FAAS |
| | | 13 Titrimetric with Fe II, persulphate oxidation |
| Mo | 1-3-4-7-8-10-13-15-17 | 6 Photometric with thiocyanate in presence of Sn II after extraction |
| | | 14 Photometric with thiocyanate in presence of ascorbic acid, after extraction |
| | | 19-20 ICP AES |
| | | 19-20 Photometric with thiocyanate without extraction |
| | | 13 Photometric with dimethylglyoxime |
| Ni | 2-3-4-7-9-10-11-12-14-15-16-17-19 | 8 FAAS |
| | | 13 ICP AES |
| | | 13 Photometric with dimethylglyoxime, after extraction |
| | | 13 Thermal conductivity, decomposition in graphite crucible |
| N | 2-4-5-6-7-8-10-11-12-13-14-15-16-17 | 3-9 Acidimetric titration after distillation, visual end point |
| | | 2-3-4-7-8-9-11-12-15-17 FAAS |
| | | 5-19 Photometric with N-benzoylphenylhydroxylamine, after extraction |
| V | 2-3-4-7-8-9-11-12-15-17 | 6-10-18 Photometric with dimethylnaphthidine |
| | | 13-14 Titrimetric with Fe II, Mn VII oxidation |
| | | 16 ICP AES |
| | | 20 Photometric with hydrogen peroxide |
| | | 16 ICP AES |

Abbreviations:-

FAAS: Flame Atomic Absorption Spectrometry

ICP-AES: Inductively Coupled Plasma Atomic Emission 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, 389 Chiswick High Road, London W4 4AL).

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 Europäischen Zertifizierten Referenzmaterialien (EURONORM-ZRM) sowie über die Anwendungen 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, 10787 Berlin).

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EUROPÄISCHES KOMITEE FÜR EISEN-UND STAHLNORMUNG

EUROPEAN CERTIFIED REFERENCE MATERIAL (EURONORM – CRM)

**SUPPLEMENTARY CERTIFICATE OF CHEMICAL ANALYSIS
FOR “ENVIRONMENTALLY SENSITIVE” ELEMENTS**

EURONORM – CRM No. 090-1 1% CARBON STEEL

LABORATORY MEANS (4 Values)
mass content in µg/g

| Line No. | Bi | Cd | Ga | Nb | Pb | Sb | Te | Tl | Zn | Hg | Se |
|----------------------|-----------------|-----------------|--------------|-------------|--------------|-------------|-----------------|-----------------|--------------|-------|------|
| 1 | <0.02 | <0.01 | 18.12 | 3.37 | 22.10 | 6.65 | <0.10 | 0.02 | 18.00 | <0.02 | 1.03 |
| 2 | 0.03 | <0.01 | — | 3.57 | 22.33 | 7.18 | <0.10 | 0.03 | 18.16 | <0.05 | 1.65 |
| 3 | 0.04 | 0.03 | 20.34 | 3.70 | 22.50 | 7.49 | <0.10 | <0.05 | 18.60 | <0.10 | 1.68 |
| 4 | <0.06 | 0.03 | 21.12 | 3.84 | 22.86 | 7.63 | <0.20 | <0.10 | 19.32 | | <2.0 |
| 5 | 0.06 | 0.03 | 21.28 | 4.00 | 23.02 | 7.97 | <0.50 | <0.10 | 19.63 | | <2.0 |
| 6 | <0.10 | <0.10 | 22.19 | 4.32 | 23.25 | 8.59 | <0.50 | <0.10 | 20.25 | | 2.0 |
| 7 | <0.10 | <0.10 | 23.16 | 4.50 | 23.33 | 8.96 | <1.00 | <0.10 | 20.62 | | |
| 8 | <0.10 | <0.10 | 23.50 | 4.52 | 23.46 | 9.24 | <1.00 | 0.18 | 20.75 | | |
| 9 | <0.10 | 0.10 | 24.89 | 5.00 | 23.50 | 9.25 | <1.00 | <0.20 | 21.45 | | |
| 10 | <0.10 | 0.12 | 24.97 | 5.15 | 23.99 | 9.42 | <2.00 | <1.00 | 21.50 | | |
| 11 | <0.20 | 0.19 | 27.94 | 5.32 | 24.28 | 9.50 | | | 21.88 | | |
| 12 | <0.20 | <0.20 | | | 24.32 | 9.68 | | | — | | |
| 13 | | | | | 24.68 | 9.75 | | | 22.02 | | |
| 14 | | | | | 25.02 | 9.96 | | | 22.02 | | |
| 15 | | | | | 25.02 | 10.39 | | | 22.32 | | |
| 16 | | | | | — | 12.00 | | | 22.50 | | |
| 17 | | | | | 26.00 | | | | 22.88 | | |
| 18 | | | | | 26.21 | | | | 23.50 | | |
| M_M | <0.20 | <0.20 | 22.75 | 4.30 | 23.87 | 8.98 | <2.00 | <1.00 | 20.91 | | |
| s_M | | | 2.78 | 0.66 | 1.22 | 1.36 | | | 1.68 | | |
| s_w | | | 0.58 | 0.41 | 0.57 | 0.52 | | | 0.86 | | |

M_M: Mean of the laboratory means s_M: Standard deviation of the laboratory 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 outlying values. Where a “—” appears in the table it indicates that an outlying value has been omitted by either the Cochran or Grubbs Test.

Values given above in italic type are for information only.

CERTIFIED VALUES
mass content in µg/g

| | Bi | Cd | Ga | Nb | Pb | Sb | Te | Tl | Zn |
|----------------------|----------------|----------------|-------------|------------|-------------|------------|----------------|----------------|-------------|
| M_M | <0.2 | <0.2 | 22.8 | 4.3 | 23.9 | 9.0 | <2.0 | <1.0 | 20.9 |
| C(95%) | | | 2.0 | 0.4 | 0.6 | 0.8 | | | 0.9 |

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 35:1989 section 4.



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MAY 2000

PARTICIPATING LABORATORIES

AG der Dillinger Hüttenwerke, Dillingen-Saar (Germany)
 Aubert & Duval, Les Ancizes (France)
 Allvac Ltd., Sheffield (UK)
 Böhler Edelstahl GmbH, Kapfenberg (Austria)
 Bundesanstalt für Materialforschung und-prüfung (BAM), Berlin (Germany)
 Centre Technique des Industries de la Fonderie (CTIF), Sèvres (France)
 Centro Nacional de Investigaciones Metalúrgicas (CENIM), Madrid (Spain)
 Hoogovens Staal BV, IJmuiden (Netherlands)
 Imphy SA, Imphy (France)
 Luxcontrol S.A., Esch-sur-Alzette (Luxembourg)

Max Planck Institut für Eisenforschung GmbH, Düsseldorf (Germany)
 OnderzoeksCentrum voor Aanwending van Staal (OCAS) NV, Zelzate (Belgium)
 Ridsdale & Co. Ltd., Middlesbrough (UK)
 Sheffield Hallam University, Materials Research Institute, Sheffield (UK)
 SOLLAC-Florange, Florange (France)
 SOLLAC-Fos-sur-Mer, Fos-sur-Mer (France)
 Swedish Institute for Metals Research (SIMR), Stockholm (Sweden)
 Voest-Alpine Stahl Linz GmbH, Linz (Austria)

DESCRIPTION OF THE SAMPLE

This sample is available in the form of chips passing a 1700µm aperture sieve from which the fines passing a 250µm aperture sieve have been removed. It is supplied in bottles containing 100g, ECRM 090-1(C). It is also supplied in the form of 38mm dia discs, ECRM 090-1(D).

INTENDED USE & STABILITY

The chip sample, ECRM 090-1(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 stoichiometric metals or 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 contents should become discoloured (eg oxidised) due to atmospheric contamination they should be discarded.

The solid (disc) sample, ECRM 090-1(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 (eg, during preparation of the working surface).

TRACEABILITY

The traceability of this ECRM is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

METHODS USED EURONORM – CRM No. 090-1

| Element | Line Number | Methods |
|-----------|-----------------------|-------------------------------------|
| Bi | 1 | FAAS using hydride generated vapour |
| | 2-5-6-11-12 | ET-AAS |
| | 3-4-7-8-9-10 | ICP-MS |
| Cd | 1-2-3-4-5-6 | ET-AAS |
| | 7-8-9-11-12 | ICP-MS |
| | 10 | FAAS |
| Ga | 1-6 | ETAAS |
| | 3-4-5-8-9-10-11 | ICP-MS |
| | 7 | FAAS |
| Nb | 1-2-4-8-9-10-11 | ICP-MS |
| | 3-5-6-7 | ICP-OES |
| Pb | 1-2-4-5-8-17-18 | ICP-MS |
| | 3-7-9-11-12-13-14 | ET-AAS |
| | 6-10-15 | FAAS |
| Sb | 1-14 | FAAS |
| | 2-5-7-9-10-11-12 | ET-AAS |
| | 3-4-6-8-13-15-16 | ICP-MS |
| Te | 2-3-6-7-8-9 | ICP-MS |
| | 1-4-5-10 | ET-AAS |
| Tl | 1-4-5-8-9-10 | ICP-MS |
| | 2-3-6-7 | ET-AAS |
| Zn | 1-8-10-13-14-15-16-17 | FAAS |
| | 2-3-5-7-9-11-18 | ICP-MS |
| | 4-6 | ET-AAS |
| Hg | 1-2 | Cold vapour AAS |
| | 3 | ICP-MS |
| Se | 1-6 | ICP-MS |
| | 2 | FAAS using hydride generated vapour |
| | 3-4-5 | ET-AAS |

Abbreviations:-

ET-AAS: Electrothermal (Graphite Furnace) Atomic Absorption Spectrometry
 FAAS: Flame Atomic Absorption Spectrometry
 ICP-MS: Inductively Coupled Plasma - Mass Spectrometry
 ICP-OES: Inductively Coupled Plasma - Optical Emission 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, 389 Chiswick High Road, London W4 4AL).

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