

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. 186-1 SILICO - MANGANESE STEEL

LABORATORY MEANS (4 Values)

mass content in %

| Line No | C | Si | Mn | P | S | Cr | Mo | Ni | Al (Tot) | Cu |
|----------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| 1 | 0.6025 | 1.6811 | 0.8550 | 0.0204 | 0.0320 | 0.2008 | 0.0426 | — | — | 0.2612 |
| 2 | 0.6062 | 1.6925 | 0.8600 | 0.0205 | 0.0336 | 0.2030 | 0.0430 | 0.1790 | — | 0.2650 |
| 3 | 0.6068 | 1.7018 | 0.8610 | 0.0206 | 0.0338 | 0.2065 | 0.0458 | 0.1839 | 0.0122 | 0.2700 |
| 4 | 0.6072 | 1.7060 | 0.8612 | 0.0207 | 0.0341 | 0.2068 | 0.0462 | 0.1840 | 0.0122 | 0.2745 |
| 5 | 0.6075 | 1.7093 | 0.8612 | 0.0208 | 0.0342 | 0.2102 | 0.0462 | 0.1840 | 0.0135 | 0.2762 |
| 6 | 0.6075 | 1.7095 | 0.8630 | 0.0210 | 0.0348 | 0.2122 | 0.0470 | 0.1862 | 0.0140 | 0.2764 |
| 7 | 0.6082 | 1.7100 | 0.8652 | 0.0213 | 0.0349 | 0.2125 | 0.0470 | 0.1872 | 0.0140 | 0.2782 |
| 8 | 0.6082 | 1.7158 | 0.8675 | 0.0217 | 0.0353 | 0.2126 | 0.0472 | 0.1875 | 0.0144 | 0.2785 |
| 9 | 0.6082 | 1.7160 | 0.8708 | 0.0220 | 0.0353 | 0.2132 | 0.0475 | 0.1885 | 0.0144 | 0.2798 |
| 10 | 0.6085 | 1.7188 | 0.8710 | 0.0221 | 0.0354 | 0.2133 | 0.0475 | 0.1885 | 0.0145 | 0.2798 |
| 11 | 0.6098 | 1.7200 | 0.8710 | 0.0221 | 0.0356 | 0.2218 | 0.0475 | 0.1898 | 0.0145 | 0.2800 |
| 12 | 0.6100 | 1.7213 | 0.8726 | 0.0225 | 0.0357 | 0.2222 | 0.0488 | 0.1900 | 0.0148 | 0.2808 |
| 13 | 0.6106 | 1.7215 | 0.8728 | 0.0228 | 0.0358 | 0.2225 | 0.0496 | 0.1900 | 0.0152 | 0.2815 |
| 14 | 0.6110 | 1.7230 | 0.8738 | 0.0228 | 0.0358 | 0.2225 | 0.0498 | 0.1905 | 0.0152 | 0.2818 |
| 15 | 0.6115 | 1.7275 | 0.8745 | 0.0230 | 0.0360 | 0.2230 | 0.0505 | 0.1923 | 0.0153 | 0.2825 |
| 16 | 0.6120 | 1.7318 | 0.8785 | 0.0232 | 0.0360 | 0.2260 | 0.0505 | 0.1932 | 0.0155 | 0.2878 |
| 17 | 0.6132 | 1.7324 | 0.8800 | 0.0235 | 0.0361 | 0.2270 | 0.0510 | 0.1950 | 0.0155 | 0.2880 |
| 18 | 0.6162 | 1.7350 | 0.8820 | 0.0238 | 0.0362 | 0.2270 | 0.0512 | 0.1956 | | 0.2908 |
| 19 | 0.6170 | 1.7495 | 0.8847 | 0.0240 | 0.0368 | 0.2279 | 0.0513 | 0.1980 | | 0.2929 |
| 20 | 0.6175 | 1.7525 | — | 0.0243 | 0.0375 | 0.2350 | 0.0528 | 0.1992 | | 0.2950 |
| 21 | 0.6178 | — | — | 0.0245 | 0.0390 | 0.2355 | | 0.2005 | | 0.3000 |
| M_M | 0.6104 | 1.7188 | 0.8698 | 0.0223 | 0.0354 | 0.2182 | 0.0482 | 0.1901 | 0.0143 | 0.2810 |
| s_M | 0.0041 | 0.0173 | 0.0083 | 0.0014 | 0.0015 | 0.0100 | 0.0028 | 0.0056 | 0.0011 | 0.0094 |
| s_w | 0.0038 | 0.0092 | 0.0049 | 0.0005 | 0.0006 | 0.0068 | 0.0015 | 0.0046 | 0.0007 | 0.0041 |

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, $s_b = \sqrt{s_M^2 - (s_w^2 \div 4)}$

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 eliminated by either the Cochran or Grubbs Test.

CERTIFIED VALUES

Mass content in %

| | C | Si | Mn | P | S | Cr | Mo | Ni | Al (Tot) | Cu |
|----------------------|---------------|--------------|--------------|---------------|---------------|--------------|---------------|--------------|---------------|--------------|
| M_M | 0.6104 | 1.719 | 0.870 | 0.0223 | 0.0354 | 0.218 | 0.0482 | 0.190 | 0.0143 | 0.281 |
| C(95%) | 0.0019 | 0.009 | 0.004 | 0.0007 | 0.0007 | 0.005 | 0.0013 | 0.003 | 0.0006 | 0.005 |

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

This reference material was prepared and issued by:

BUREAU OF ANALYSED SAMPLES LIMITED

Newham Hall, Middlesbrough, England TS8 9EA

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, Nordic Countries – Nordic CRM Working Group; UK – BAS Ltd.)



METHODS USED
EURONORM – CRM No. 186-1

| Element | Line Number | Methods |
|-----------------|-------------------------------------|---|
| C | 1-2-11-17-19 | Combustion, coulometric |
| | 3-10-14 | Combustion, non-aqueous titration |
| | 4 | Combustion, gravimetric |
| | 5-12-16 | Combustion, conductimetric |
| | 6-9 | Combustion, thermal conductivity |
| | 7-8-13-15-18-20-21 | Combustion, infrared absorption |
| Si | 1 | Gravimetric, dehydration with sulphuric acid |
| | 2-12 | Atomic absorption spectrometry |
| | 3-4-5-7-8-10-11-13-16-17-18-19 | Gravimetric, dehydration with perchloric acid |
| | 6-9 | Photometric as molybdenum blue |
| | 14 | Gravimetric, dehydration with hydrochloric acid |
| | 15 | Titrimetric as potassium fluosilicate |
| Mn | 20 | Photometric, as molybdenum blue, with extraction |
| | 1-3-6-9-10-11-14-15-17 | Photometric, oxidation with periodate |
| | 2 | Titrimetric with ammonium ferrous sulphate, zinc oxide separation |
| | 4-5-8-12-13-19 | Atomic absorption spectrometry |
| | 7 | Titrimetric with arsenite, oxidation with persulphate/silver nitrate |
| | 16-18 | Photometric, oxidation with persulphate/silver nitrate |
| P | 1-4-5-7-9-11-18 | Photometric as molybdenum blue |
| | 2-6-8-10-15-16-21 | Photometric as phosphovanadomolybdate with extraction |
| | 3 | Photometric as molybdenum blue, with extraction |
| | 12-17 | Photometric as phosphovanadomolybdate |
| | 13-14-19-20 | Titrimetric as phosphomolybdate |
| S | 1-7 | Combustion, oxidation/reduction titration |
| | 2-13-21 | Combustion, conductimetric |
| | 3-4-6-10-14-15-16-17-18-20 | Combustion, infrared absorption |
| | 5-8-11-12 | Gravimetric, as barium sulphate |
| | 9 | Combustion, acidimetric titration |
| | 19 | Combustion, coulometric |
| Cr | 1-6-8-10-11-14-15-16-17-18-19-20-21 | Atomic absorption spectrometry |
| | 2-5-7-12-13 | Titrimetric with ammonium ferrous sulphate, oxidation with persulphate/silver nitrate |
| | 3-4-9 | Photometric with diphenylcarbazide |
| Mo | 1-3-4-5-9-20 | Photometric as thiocyanate with extraction |
| | 2-6-7-14-15-16-18 | Atomic absorption spectrometry |
| | 8-10-11-12-13-17-19 | Photometric as thiocyanate |
| Ni | 2-3-5-9-11-13-14-15-16-18-21 | Atomic absorption spectrometry |
| | 4-7-10-12-17-19-20 | Photometric with dimethylglyoxime |
| | 6 | Dimethylglyoxime precipitation, titration with cyanide |
| | 8 | Dimethylglyoxime precipitation, titration with EDTA |
| Al (Tot) | 3-4-5-6-8-9-10-11-14-15-16-17 | Atomic absorption spectrometry |
| | 7 | Photometric with eriochrome cyanine |
| | 12 | Photometric with chrome azurol S |
| | 13 | Photometric with chrome azurol S, ion exchange separation |
| Cu | 1 | Photometric with biscyclohexanone oxalyldihydrazone |
| | 2 | Iodimetric, separation as thiocyanate |
| | 3-6-7-8-9-10-12-13-14-16-17-19 | Atomic absorption spectrometry |
| | 4 | Iodimetric, separation as sulphide |
| | 5 | Photometric with dithiooxamide |
| | 11-20 | Photometric with diethyldithiocarbamate |
| | 15 | Photometric with 2,2' diquinolyl |
| | 18 | Photometric as ammine complex |
| | 21 | Polarography |

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DESCRIPTION OF THE SAMPLE

The sample is available in the form of chips passing a 1700 µm aperture sieve from which the fines passing a 250 µm aperture sieve have been removed. It is supplied in bottles containing 100 g...ref ECRM 186-1(C). It is also supplied in the form of 38 mm dia. discs...ref ECRM 186-1(D).

INTENDED USE & STABILITY

The chip sample, ECRM 186-1 (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 contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

The disc sample, ECRM 186-1 (D), is intended for establishing and checking the calibration of Optical Emission and X-Ray Spectrometers for the analysis of similar materials. The "as received" working surface of the sample should be finished before use to remove any protective coating. The disc will remain stable provided that it is not subject to excessive heat (e.g. during preparation of the working surface).

TRACEABILITY

The traceability of ECRM 186-1 has been established in accordance with principles of ISO Guides 30 – 35 and the International vocabulary of basic and general terms in metrology.

The characterisation of this material has been achieved by inter-laboratory study, 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.

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 CEN Report CR 10317 and ECISS Information Circular No. 5, both of which are available from the national standards body in your country or from CEN in Brussels. (In the UK this is the BSI, 389 Chiswick High Road, London W4 4AL).

Further information and advice on this or other Certified Reference Materials or Reference Materials may be obtained from the address below.

Pour disposer d'informations 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 ce certificat, se reporter soit au producteur de ce Matériau de Référence Certifié, à l'adresse figurant sur ce Certificat soit au Rapport CEN CR 10317 et à la circulaire d'information No. 5 (ECISS). On peut se procurer ces deux documents auprès des organismes nationaux de normalisation ou auprès du CEN, Bruxelles. (Pour la France: AFNOR, 11 rue Francis de Pressensé, 93571 La Plaine St Denis cedex).

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REVISION HISTORY

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Revised with C(95%) and s_w values for each certified element and improved accuracy for C, Si, P, S, Mo & Al

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