

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. 579-1 FERRO-NIOBIUM**

**LABORATORY MEANS (4 Values)**  
**mass content in %**

| Line No              | C             | Si           | P             | S             | Al           | Co            | Nb           | Sn            | Ti            | Ta           |
|----------------------|---------------|--------------|---------------|---------------|--------------|---------------|--------------|---------------|---------------|--------------|
| 1                    | 0.0342        | 0.938        | 0.0560        | 0.0186        | 1.740        | 0.0035        | —            | —             | 0.4950        | 3.619        |
| 2                    | 0.0345        | 0.962        | 0.0573        | 0.0188        | 1.778        | 0.0036        | 62.57        | 0.3150        | 0.5150        | 3.632        |
| 3                    | 0.0350        | 0.970        | 0.0580        | 0.0192        | 1.785        | 0.0040        | 62.63        | 0.3250        | 0.5150        | 3.712        |
| 4                    | 0.0350        | 0.979        | 0.0595        | 0.0198        | 1.798        | 0.0042        | 62.66        | 0.3275        | 0.5312        | 3.725        |
| 5                    | 0.0350        | 1.000        | 0.0624        | 0.0200        | 1.828        | 0.0045        | 62.78        | 0.3300        | 0.5365        | 3.742        |
| 6                    | 0.0356        | 1.003        | 0.0625        | 0.0202        | 1.830        | 0.0046        | 62.78        | 0.3338        | 0.5365        | 3.802        |
| 7                    | 0.0356        | 1.030        | 0.0648        | 0.0204        | 1.840        | 0.0050        | 62.80        | 0.3400        | 0.5385        | 3.812        |
| 8                    | 0.0362        | 1.040        | 0.0658        | 0.0205        | 1.840        | 0.0052        | 62.80        | 0.3418        | 0.5385        | 3.816        |
| 9                    | 0.0368        | 1.042        | 0.0660        | 0.0210        | 1.842        | 0.0056        | 62.85        | 0.3445        | 0.5416        | 3.832        |
| 10                   | 0.0369        | 1.049        | 0.0662        | 0.0212        | 1.855        | 0.0058        | 62.86        | 0.3454        | 0.5425        | 3.842        |
| 11                   | 0.0370        | 1.057        | 0.0678        | 0.0213        | 1.855        | 0.0058        | 62.87        | 0.3490        | 0.5650        | 3.885        |
| 12                   | 0.0371        | 1.061        | 0.0678        | 0.0216        | 1.878        | 0.0058        | 62.91        | 0.3550        | 0.5778        | 3.895        |
| 13                   | 0.0372        | 1.080        | 0.0682        | 0.0218        | 1.911        | 0.0060        | 63.00        | 0.3575        | 0.6162        | 3.942        |
| 14                   | 0.0375        | 1.088        | 0.0691        | 0.0220        | 1.912        | 0.0060        | 63.00        | 0.3620        | 0.6274        | 3.992        |
| 15                   | 0.0375        | 1.105        |               | 0.0222        | 1.918        | 0.0068        | 63.04        | 0.3675        | 0.6425        | 4.015        |
| 16                   | 0.0379        | 1.125        |               | 0.0230        | 1.942        |               | 63.08        | 0.3691        | 0.6550        | 4.046        |
| 17                   | 0.0385        |              |               | 0.0242        | 1.953        |               | 63.21        |               | 0.6600        | 4.198        |
| 18                   | 0.0385        |              |               |               | 1.968        |               |              |               |               |              |
| 19                   | 0.0395        |              |               |               |              |               |              |               |               |              |
| <b>M<sub>M</sub></b> | <b>0.0366</b> | <b>1.033</b> | <b>0.0637</b> | <b>0.0209</b> | <b>1.860</b> | <b>0.0051</b> | <b>62.87</b> | <b>0.3442</b> | <b>0.5667</b> | <b>3.853</b> |
| <b>S<sub>M</sub></b> | <b>0.0015</b> | <b>0.054</b> | <b>0.0045</b> | <b>0.0015</b> | <b>0.065</b> | <b>0.0010</b> | <b>0.18</b>  | <b>0.0162</b> | <b>0.0530</b> | <b>0.153</b> |

M<sub>M</sub>: Mean of the intralaboratory means, S<sub>M</sub>: Standard deviation of the intralaboratory means

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" values by the participating laboratories have not been taken into account in the statistical calculations.

**CERTIFIED VALUES**

**Mass content in %**

|                      | C            | Si          | P            | S            | Al          | Co            | Nb           | Sn           | Ti           | Ta          |
|----------------------|--------------|-------------|--------------|--------------|-------------|---------------|--------------|--------------|--------------|-------------|
| <b>M<sub>M</sub></b> | <b>0.037</b> | <b>1.03</b> | <b>0.064</b> | <b>0.021</b> | <b>1.86</b> | <b>0.0051</b> | <b>62.87</b> | <b>0.344</b> | <b>0.567</b> | <b>3.85</b> |
| <b>C(95%)</b>        | <b>0.001</b> | <b>0.03</b> | <b>0.003</b> | <b>0.001</b> | <b>0.04</b> | <b>0.0006</b> | <b>0.10</b>  | <b>0.009</b> | <b>0.028</b> | <b>0.08</b> |

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

**PARTICIPATING LABORATORIES**

|  |  |
|--|--|
| Arbed, d'Esch-Belval, Esch-sur-Alzette, Luxembourg<br>Aubert et Duval, Les Ancizes, France<br>Böhler AG, Düsseldorf 11, Germany<br>British Steel Corporation, Consett Works, U.K.<br>British Steel Corporation, Scunthorpe Works, U.K.<br>Bundesanstalt für Materialprüfung (BAM), Berlin-Dahlem, Germany<br>Cockerill, Seraing, Belgium<br>ESTEL-Hoogovens BV, IJmuiden, Holland<br>Gesellschaft für Elektrometallurgie, Nürnberg, Germany<br>IRSID, Maizières-les-Metz, France<br>IRSID, St Germain-en-Laye, France<br>Laboratoire de Firminy, Creusot Loire, France | Krupp Stahl AG, Bochum, Germany<br>London & Scandinavian Metallurgical Co. Ltd., Rotherham, U.K.<br>Murex Ltd., Rainham, U.K.<br>Ridsdale & Co. Ltd., Middlesbrough, U.K.<br>SACILOR, Florange, France<br>SNIAS, Suresnes, France<br>SOFREM, Le Fayet, France<br>Sollac, Florange, France<br>Stahlwerke Röchling-Burbach, Völklingen-Saar, Germany<br>Thyssen Edeistahlwerke AG, Krefeld, Germany<br>Usinor, Dunkerque, France |
|--|--|

**This Certified Reference Material was prepared in accordance with the principles and recommendations set out in ISO Guides 30 – 35 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: Stahlinstitut VDEh, BAM Bundesanstalt für Materialforschung und –prüfung & MPI für Eisenforschung, Nordic Countries – Nordic CRM Working Group, UK – BAS Ltd.)

# EURONORM – CRM No. 579-1

## METHODS USED

| Element   | Line Number                         | Methods   |
|-----------|-------------------------------------|---|
| <b>C</b>  | 1-15                                | Combustion, non-aqueous titration after absorption in organic solvent                                   |
|           | 2-3-4-6-7-8-10-11-12-13-14-16-17-19 | Combustion, infrared absorption   |
|           | 5-9                                 | Combustion, thermal conductivity  |
|           | 18                                  | Combustion, coulometric titration   |
|           |                                     |   |
| <b>Si</b> | 1-2-3-5-6-8-9-10-11-12              | Gravimetry, dehydration with sulphuric acid   |
|           | 4                                   | Spectrophotometry, silicovanadomolybdate  |
|           | 7-13-14-15                          | X-ray fluorescence spectrometry   |
|           | 16                                  | Atomic absorption spectrometry  |
| <b>P</b>  | 1-2-5-7-8-10                        | Spectrophotometry, phosphovanadomolybdate, extraction   |
|           | 3-11                                | X-ray fluorescence spectrometry   |
|           | 4-12                                | Spectrophotometry, molybdenum blue, extraction  |
|           | 6-9-13                              | Spectrophotometry, molybdenum blue without extraction   |
|           | 14                                  | Spectrophotometry, molybdenum blue, chromatographic separation  |
| <b>S</b>  | 1-2-3-4-5-6-9-11-14-15-16-17        | Combustion, infrared absorption   |
|           | 7                                   | Combustion, oxidation/reduction titration   |
|           | 8-10                                | Combustion, acidimetric titration   |
|           | 12                                  | Combustion, spectrophotometry, para rosaniline  |
|           | 13                                  | Combustion, conductimetry   |
| <b>Al</b> | 1-3-4-7-8-9-10-11-18                | Atomic absorption spectrometry  |
|           | 2                                   | Gravimetry as hydroxyquinolate  |
|           | 5-6-13-16                           | X-ray fluorescence spectrometry   |
|           | 12-14-17                            | Atomic absorption spectrometry, after separation  |
|           | 15                                  | Spectrophotometry, 8-hydroxyquinoline, with extraction  |
| <b>Co</b> | 1-2-3-6-7-8-9-12-13-14-15           | Atomic absorption spectrometry  |
|           | 4                                   | Spectrophotometry with nitroso-R-salt, separation with 1-nitroso-2-naphthol                             |
|           | 5-10                                | Spectrophotometry, nitroso-R-salt   |
|           | 11                                  | Spectrophotometry, 4-(5-chloropyridyl-2-azo)-m-phenylenediamine, chromatographic separation             |
| <b>Nb</b> | 2-3-6-8-9-11-14                     | X-ray fluorescence spectrometry   |
|           | 4                                   | Atomic absorption spectrometry  |
|           | 5-10-15-16                          | Gravimetry as Nb <sub>2</sub> O <sub>5</sub> , precipitation with cupferron, chromatographic separation |
|           | 7                                   | Spectrophotometry, PAR, chromatographic separation  |
|           | 12-13                               | Gravimetry as Nb <sub>2</sub> O <sub>5</sub> , precipitation by hydrolysis                              |
|           | 17                                  | Gravimetry as Nb <sub>2</sub> O <sub>5</sub> , precipitation with phenylarsonic acid                    |
| <b>Sn</b> | 2-3-6-7-9-11-12-13-15-16            | Atomic absorption spectrometry  |
|           | 4-5                                 | X-ray fluorescence spectrometry   |
|           | 8                                   | Iodimetric titration, separation of niobium by hydrolysis   |
|           | 10                                  | Spectrophotometry, phenylfluorone, halide separation  |
|           | 14                                  | Iodimetric titration, sulphide separation   |
| <b>Ti</b> | 1-3-12-17                           | X-ray fluorescence spectrometry   |
|           | 2-5-6-11-14                         | Atomic absorption spectrometry  |
|           | 4                                   | Spectrophotometry, hydrogen peroxide, chromatographic separation  |
|           | 7                                   | Spectrophotometry, dantipyrylmethane, chromatographic separation  |
|           | 8-9-16                              | Spectrophotometry, chromotropic acid, chromatographic separation  |
|           | 10                                  | Spectrophotometry, dantipyrylmethane  |
|           | 13-15                               | Spectrophotometry, hydrogen peroxide  |
| <b>Ta</b> | 1                                   | Spectrophotometry, methyl violet  |
|           | 2-3-5-6-7-11-14-16                  | X-ray fluorescence spectrometry   |
|           | 4                                   | Spectrophotometry, pyrogallol   |
|           | 8-9-12-15                           | Gravimetry, chromatographic separation and precipitation with cupferron                                 |
|           | 10-13                               | Spectrophotometry, phenylfluorone, chromatographic separation   |
|           | 17                                  | Gravimetry, precipitation with phenylarsonic acid, separation from niobium by extraction                |

## DESCRIPTION OF THE SAMPLE

The sample consists of a powder with a nominal particle size of 53 – 150µm. It is supplied in bottles containing 100g.

## INTENDED USE & STABILITY

ECRM 579-1 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 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.

## TRACEABILITY

The traceability of ECRM 579-1 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 either to the producer of this Certified Reference Material or to Technical Reports CEN/TR 10317:2013 and CEN/TR 10350:2013, 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).

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd. 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, soit aux Rapports Techniques CEN/TR 10317:2013 et CEN/TR 10350:2013. On peut se procurer ces deux documents auprès des organismes nationaux de normalisation. (Pour la France: AFNOR, 11 Avenue Francis de Pressensé, 93571 – St Denis la Plaine Cedex).

D'autres informations et avis au sujet de ce Matériau de Référence Certifié, ou de tout autre Matériau de Référence Certifié ou Matériau de Référence produits par le Bureau of Analysed Samples Ltd., peuvent être demandés en contactant l'adresse figurant dans le bas de ce Certificat.

Angaben über Herstellung, Zertifizierung und Bezugsmöglichkeiten dieser Europäischen Zertifizierten Referenzmaterialien (EURONORM-ZRM) sowie über die Anwendungen der in diesem Zertifikat enthaltenen statistischen Daten sind erhältlich beim Hersteller dieses zertifizierten Referenzmaterials, oder sie finden sich in den CEN-Reports CEN/TR 10317:2013 und CEN/TR 10350:2013, beide zu beziehen durch die nationalen Normenorganisationen. (In Deutschland bei der Vertriebsstelle des DIN: Beuth-Verlag GmbH, Burggrafenstraße 4-10, 10787 Berlin).

Weitere Informationen und Hinweise zu diesem oder anderen durch Bureau of Analysed Samples Ltd. hergestellten zertifizierten Referenzmaterialien oder Referenzmaterialien können unter der unten angegebenen Adresse erhalten werden.

För information angående tillverkning, certifiering och anskaffning av dessa europeiska certifierade referensmaterial (EURONORM CRM) och för användning av statistisk information, som angivits i detta certifikat, refereras antingen till producenten av detta certifierade referensmaterial eller till Teknisk Rapport CEN/TR 10317:2013 och CEN/TR 10350:2013 som kan erhållas från den nationella standardiseringsorganisationen. (Sverige: SIS, S:t Paulsgatan 6, SE-118 80 Stockholm, Finland: SFS, PL. 116, FIN-002 41, Helsingfors, Danmark: DS, Kollegievej 6, DK-Charlottenlund 2920, Norge: NSF, Drammensveien, 145 A, Postboks 353 Skøyen, NO-0213 Oslo, Island: STRI, Holtagardar, IS-104 Reykjavik).

Ytterligere informasjon og rådfrågan om detta eller andra Certifierade Referensmaterial/Referensmaterial, producerade av Bureau of Analysed Samples Ltd. kan erhållas från angiven adress enligt nedan.

NEWHAM HALL, NEWBY,  
MIDDLESBROUGH, ENGLAND, TS8 9EA  
Email: enquiries@basrid.co.uk  
Website: www.basrid.co.uk

For BUREAU OF ANALYSED SAMPLES LTD.

R P MEERES,  
MANAGING DIRECTOR