# **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. 097-1 HIGH PURITY IRON

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

S	TO 100 100 100 100 100 100 100 100 100 10									
Line No.	Mn	Р	8	Cr	NI	As	8	Co	Cu	N
1	57	12	18	10	22 23	44	2	30	17	5
2	58	13	20	11	23	45	2	32	18	6
8	58	14	20	11	23	45	2	34	18	6
4	59	14	21	12	24 .	48	2	34	19	6
5	60	14	21	12	24	48	2	34	19	6
6	61	15	21	12	24	48	2	35	20	7
7	62	16	21	14	24	50	2	35	20	7
à	64	16	21	14	25	- 51	2	35	20	7
9	64	16		16	25 25	51	·2	35 35	20 20 20 20 20	7
10	65	16	21 22 23 23	16	25 25	51	2	85	20	8
-11	85	17	23	18	25	52	. 2	35	20 20 20 20 20 21	8
12	66	17	23	18	26	52 53	3	36	20	8
12 13	66	17	23	18	26	53	3	37	20	8
14	66	17	23	18	27	54·	3	37	21	8
15	66	18	24	18	28	54	3	37	21	9
16	66	18	24	18	30	57	4	38	21	9
17	67	18	24	20	30 32	60	4	39	22	
18	68	18	25	28		61	4	40	24 26	
19	68	18	25 26 26	24	_		4	43	26	
20	73	20	26	25			4	48	1 <del>-</del>	
21	i a	22		10			_	_		
MM	64	16	22	16	25	51	8	37	20	7
s <sub>M</sub>	4	3	2	5	3	6	1 1	4	2	1 1
8 <sub>W</sub>	2	2	1	2	2	8	1	2	1	1
-W	_ Z	1 2				1				

M<sub>M</sub>: Mean of the intralaboratory means <sup>a</sup>m: Standard deviation of the Intralaboratory means <sup>a</sup>m: Intralaboratory standard deviation <sup>a</sup>b: Interlaboratory standard deviation

 $s_{M} = \sqrt{s_{b}^{2} + s_{W}^{2}/4}$ 

The laboratory mean values have been examined statistically to eliminate any outlying values. Where a "-" appears in the table it indicates that an outlying value has been omitted.

# CERTIFIED VALUES mass content in µg/g

	Mn	P	S	Çr	Ni	As	В	Co	Сı	N
MM	64	16	22	16	25	51	3	37	20	7
s <sub>M</sub>	4	3	2	5	3	5	1	4	2	1

NB: This sample has been found to be inhomogeneous with respect to aluminium and therefore the previously certified value for this element has been withdrawn.

#### **DESCRIPTION OF THE SAMPLE**

This sample is available in the form of chips all passing a 1700  $\mu$ m aperture sleve from which the dust passing a 250  $\mu$ m aperture sleve has been removed, it is supplied in bottles containing 100g ....ref 097-1(C). It is also supplied in the form of 38mm dia. discs ....ref 097-1(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: German Federal Republic-Iron and Steel CRM Working Group VDEh, BAM & MPI für Eisenforschung; UK-BAS Ltd.)

Revised FEBRUARY 1998

(First issued in November 1984 without certified values for B and before re-certification of P, then revised in February 1990)

#### **PARTICIPATING LABORATORIES**

AB Sandvik Steel, Sandviken (Sweden)

AG der Dillinger Hüttenwerke, Dillingen-Saar (West Germany)

AERE Hanwell, Environmental and Medical Science Division, Abingdon (UK)

ARBED, Division d'Esch-Belval, Esch-sur-Alzette (Luxembourg)

British Steel, Ravenscralg Works, Motherwell (UK)

British Steel Technical, Corby (UK)

British Steel Technical, Port Taibot (UK)

British Steel Technical, Swinden Laboratories, Rotherham (UK)

Bundesanstalt für Materialforschung und prüfung (BAM) Berlin (West Germany)

Centro Sperimentale Metallurgico (CSM), Rome (Italy)

Cockerlii Sambre SA, Couillet (Belgium)
Cockerlii Sambre SA, Seraing (Belgium)
Hoesch Stahl AG, Dortmund (West Germany)

Hoogovens Groep BV, Umulden (Netherlands)

Institut de Recherches de la Sidérurgie Française (França)

Krupp Stahl AG, Bochum (West Germany)

Régle Nationale des Usines Renault (RNUR) Billancourt (France)

Ridsdale and Co Ltd, Middlesbrough (UK) Rotherham Engineering Steels, Rotherham (UK) SKF Steel Hellefors AB, Hällefors (Sweden)

Soc Terni, Terni (Italy) SOLLAC, Florange (France)

SOLMER, Fos-sur-Mer (France)

Stahlwerke Peine-Salzgitter, Salzgitter (West Germany)

Stocksbridge Engineering Steels, Sheffield (UK) Svensk Stahl Domnarvet, Borlange (Sweden) Thyssen Stahl AG, Dulsburg (West Germany)

Usinors Aciers, Dunkerque (France)

# APPROXIMATE VALUES FOR INFORMATION mass content in pg/g

Line No.	Si (Total)	Мо	Nb	Pb	Sn	π	ν	<b>Z</b> r	Bi	Ċa	Sb
1 2 3 4 5 6 7 8 9 10 11 2 13 14 15 16 17 8 19 2 21	37 45 49 50 51 52 80 74 75 77 79 80 98	1 1 1 1 1 2 2 2 3 4 4 4 5 5 6 6 8 0 0 0 V V	<pre>&lt; 1   &lt; 2   &lt; 3   &lt; 5   &lt; 10   &lt; 10   &lt; 10   </pre>	0.3 0.4 0.8 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.1 0.1	5 6 7 7 8 8 8 8 4 10 11 12 13 18 21	V 1 V 1 2 2 2 3 V 8 10 11 12	1 1 2 2 3 3 5 5 5 5 5 5 10 0	1 2 2 2 9 9	<1 1 1 <2 2	<pre>&lt;1 &lt;1 &lt;1 22333 </pre>	9 10 10 10
Content	< 100	< 10	< 10	<5	< 25	< 15	< 10	< 10	<5	<5	≤10

Additional approximate values in  $\mu g/g$ :- C\*< 5, O = 500, Mg < 5, Ta < 5, W ≤ 10, Zn <1

\*Note: The carbon content of the chip sample, ECRM 097-1(C), has not been certified because the surface of this material has found to be slightly contaminated with carbonaceous matter.

The carbon content of the disc material has now been certified and details are given in the Supplementary Certificate dated February 1998, issued with ECRM 097-1(D).

# METHODS USED EURONORM - CRM 097-1

	EURONOAM	
Element	Line Number	Methods
Ma	2-4-5-6-8-9-13-14-17-20 3 11-18	Fhotometric, oxidation with periodate  AAS PES AAS, after separation of iron PES, after ion exchange separation
P	4-16 5-7-10-15-19	Photometric as phosphovanadomolybdate with extraction Photometric as molybdenum blue with extraction Photometric as molybdenum blue without extraction PES
S	3-11 13	Combustion, Infrared absorption Combustion, conductivity Gravimetric as barium sulphate after chromatographic separation on alumina Combustion, coulometric titration Combustion, acidimetric titration
Cr .	13	
NI	6-14 12	AAS PES AAS, after separation of Iron PES, after ion exchange separation Photometric with dimethylglyoxime, with extraction
As	4-10-12-16 5-11-13-15 6-7 14	Photometric as molybdenum blue, halide extraction AAS-ETA Photometric with silver diethyldithlocarbamate, separation as arsine AAS-evolution as arsine AAS PES
В	3-5-6-12-17-18 4-10 9 13 16	Photometric with curcumin, EN 10200 Photometric with curcumin Photometric with 1-1 dianthrimide, with separation PES Photometric with quinalizarin Photometric with Nile Blue A Photometric as methylene blue fluoborate, with extraction
જ	3-9 12	AAS Photometric with nitroso-R-salt AAS after separation of iron PES PES, after ion exchange separation
Cu	2-3 <del>-4-6-8</del> -10-11-12-13-14-16-17-18-19 5 7	Photometric with neocuproine, with extraction AAS PES, after ion exchange separation Photometric with diethyldithlocarbamate, with extraction AAS, after separation of Iron
N	6-12	Thermal conductivity, decomposition in graphite crucible Manometry, vacuum fusion Photometric with indophenol blue, separation by distillation
Si (total) Mo	1-2-4-5-9-10-11-12 8 3-6-7-13 1-4-5-9-13-14-16	<u> </u>
, NAC	2- <del>6</del> -7-19 3 8-10-12-17 11-18	

#### METHODS USED EURONORM - CRM 097-1

Element	Line Number	Methods
Nb	2-3 4 5-7-10 6-8	Photometric with PAR
Pb	1-2-3-6-7-9 4-8 5-10-11-13	AAS, extraction with TOPO AAS
Sn	1-2-3-4-13 5 6-7-10 8 9 11	AAS, evolution as hydride AAS-ETA Photometric with pyrocatechol violet lodometric titration, reduction with Al Photometric with fluorone, halide separation
77	14 1-9 2 3-7-10 4 5	Photometric with chromotropic acid, after ion exchange separation PES, after ion exchange separation
v	8 11 12 1-9 2 3-8 4-11	XRF, fused bead technique on the isolated oxides AAS Photometric with chromotropic acid. Photometric with diantipyrylmethane AAS-ETA PES, after ion exchange separation PES Photometric with N-benzoylphenyl hydroxylamine, with extraction AAS, after separation of iron
Zr	6 7-10-12-13 1 2-4 3	XRF, fused bead technique on the isolated oxides
Bi	1-2-	AAS-ETA AAS, after separation of iron
Ca	1-2-3-4-5-6-7-8-9-10 11	AAS AAS, after separation of iron
Sb	1-3-4-5 2	AAS AAS-ETA

#### Abbreviations:-

AAS : Atomic Absorption Spectrometry

AAS-ETA : Atomic Absorption Spectrometry—Electrothermal

Atomisation

PAR : 4—(2 pyridylazo) resorcinol
PES : Plasma Emission Spectrometry
TOPO : Tri-octylphosphine oxide
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, 389 Chiswick High Road, London W4 4AL).

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EUROPEAN CERTIFIED REFERENCE MATERIAL (EURONORM - CRM)

# SUPPLEMENTARY CERTIFICATE OF CHEMICAL ANALYSIS FOR CARBON EURONORM – CRM No. 097-1 (D) HIGH PURITY IRON

These values are valid only for the solid form of the sample

#### **LABORATORY MEANS (4 Values)**

Line No.		SAMPLE		CARBON CONTENT	COMB		
	Form	Pretreatment	<b>W</b> t (g)	INTERNAL (µg/g)	Туре	Temp °C	DETECTION
1	С	Etched HF/HNO <sub>3</sub>	0.35	0.50	HF		Infrared
2	l c	Etched HF/H <sub>2</sub> O <sub>2</sub>	0.2	0.72	HF	1450	Infrared
3	C	Etched HF/H <sub>2</sub> O <sub>2</sub>	0.90	1.50	HF		Infrared
4	] C	Etched HF/H <sub>2</sub> O <sub>2</sub>	0.8-1.3	1.60	R	1250	Coulometric
5	С	Heated at 400°C 5 min.		1.63	R	1250	Infrared
6	P	Surface milled/Ether washed	1.0	2.24	R	1350	Infrared
7	Р	Heated at 410°C, 10 min.		3.00	HF	1	Intrared
8	P	Hot boat at 600°C		3.00	HF	1	infrared
9	P	Heated at 420°C, 10 min.	0.9-1.1	3.01	HF	1	Infrared
10	P	Heated at 420°C, 10 min.	1.0	3.60	HF	1	Infrared
11	С	Heated at 400°C	1.3-2.0	3.67	HF	ŀ	Conductimetrk
12	P	Hot crucible	1.0	3.77	HP		Infrared
13	P	Surface ground	0.7-0.8	3.85	HF	<b>!</b>	Infrared
			MM	2.47	<del></del>	<del></del>	*
			s <sub>M</sub>	1.17			
			s <sub>w</sub>	0.52			

MM: Mean of the laboratory means SM: Standard deviation of the laboratory means Sw: Intralaboratory standard deviation

 $s_{M} = \sqrt{s_{b}^{2} + s_{w}^{2}/4}$ 

Notes:- SAMPLE Form: "C" Cut; "P" Punched; COMBUSTION; "HF" High Frequency Induction Furnace; "R" Resistance Furnace

# CERTIFIED CARBON VALUE (see "Explanatory Note" on third page) mass content in µg/g

MM	2.5		
C(95%)	0.7		

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 carbon determinations use only the sample supplied in 38mm bar form, either as 25/30mm thick discs or as 3mm thin discs suitable for cutting or punching.

#### **PARTICIPATING LABORATORIES**

AG Der Dillinger Hüttenwerke, Dillingen (Germany)
British Steel Engineering Steels, Stocksbridge (UK)
British Steel Teesside Laboratories, Middlesbrough (UK)
British Steel Strip Products, Lianwern Works, Newport (UK)
British Steel Strip Products, Port Talbot Works, Port Talbot (UK)
Hoogovens Staal BV, Urnulden (Netherlands)
Institut für Festkörper-und Werkstofforschung, Dresden (Germany)

iRSID, Malzières-les-Metz (France)
Materialprüfungsamt Nordmein Westfalen, Dortmund (Germany)
Max-Plenck Institut für Metallforschung, Institut für
Werkstoffwissenschaft, Stuttgart (Germany)
SOLLAC, Dunkerque (France)
SSAB Oxelösund, Oxelösund (Sweden)
Thyssen Stahl AG, Duisburg (Germany)



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Group, VDEh, BAM & MP! für Elsenforschung; UK-BAS Ltd.)

FEBRUARY 1998

Supplement to Certificate dated February 1990 which superseded the original certificate dated November 1984

# ADDITIONAL INFORMATION SUPPLIED BY THE PARTICIPATING LABORATORIES FOR CARBON IN ECRM 097-1 (D)

- Line 1: See References (1), (2) and (3) in the Explanatory Note. Calibrated with a mixture of carbon dioxide and oxygen gases. This value includes surface carbon which is approximately 0.2 µg/g.
- Line 2: Procedure as per reference (2) in the Explanatory Note. Calibrated with mixtures of carbon dioxide and nitrogen gases.
- Line 3: Calibrated with a mixture of carbon dioxide and oxygen gases. Reported values obtained using hot crucible after etching with hydrogen peroxide and hydrofluoric acids. Additional determinations using a cold crucible after etching gave a mean value of 2.1 μg/g C. Determinations without etching treatment of the sample gave a mean value of 3.7 μg/g C.
- Line 4: Calibrated with calcium carbonate.

The etched sample was washed and dried and then placed in a hot (250°C) combustion boat before inserting into the furnace. See Reference (5) in the Explanatory Note.

Line 5: Calibrated with sucrose.

A similar mean value of 1.60 μg/g C was obtained on cut samples without preheating at 400°C before combustion. Punched samples however gave higher mean values of 2.57 μg/g C without preheating and 1.94 μg/g C after preheating. The chip sample of ECRM 097-1, preheated at 400°C, gave a mean value of 3.98 μg/g C. All results were obtained in an analysis time of 80 seconds.

Line 6: Calibrated with sucrose.

Additional punched samples cleaned mechanically with a tungsten carbide milling machine gave a mean value of 3.33  $\mu$ g/g C. Punched samples, preheated for 5 seconds prior to analysis by HF-IR, gave a mean value of 2.75  $\mu$ g/g C. Chip samples, analysed under similar conditions to that used for the punched samples, gave a mean value of 5.1  $\mu$ g/g C (s.d. 1.1  $\mu$ g/g C) on 10 samples without pretreatment and a mean value of 2.6  $\mu$ g/g C (s.d. 0.5  $\mu$ g/g C) on 12 samples after preheating 450°C for 15 minutes.

- Line 7: Calibrated with sucrose.
- Line 8: The punched sample was cleaned in ether and placed in a hot combustion boat at approximately 600°C immediately before combustion. Using the same technique, freshly prepared millings gave a mean value of 6.0 μg/g C. Chips from a previously unopened bottle of ECRM 097-1 gave a mean value of 3.9 μg/g C.
- Line 9: Calibrated with sucrose.

Portions of the chip sample of ECRM 097-1 were also analysed under the same conditions as the punched samples. They were preheated at 420°C for 10 minutes immediately before combustion. A mean value of 3.03 µg/g C was obtained.

Line 10: The pretreatment of the punched samples at 420°C was carried out in a stream of oxygen instead of air to eliminate the risk of contamination.

Additional determinations on 4 punched samples preheated at 420°C for 10 minutes were carried out on a second HF-iR analyser. These gave a mean value of 3.7  $\mu g/g$  C.

Further determinations on 3 untreated punched samples gave a mean value of 4.2 µg/g C.

Some determinations were also carried out on chip samples of ECRM 097-1 with the following results:-

Untreated chips 5.8  $\mu$ g/g C (mean of 5 samples) Chips preheated at 420 °C for 10 minutes 3.6  $\mu$ g/g C (mean of 10 samples) Chips etched with HF/H<sub>2</sub>O<sub>2</sub> 3.6  $\mu$ g/g C (mean of 5 samples)

Pretreatment with HF/H<sub>2</sub>O<sub>2</sub> is not recommended for chip samples because of the vigorous reaction; only for solid samples.

A resistance furnace was coupled to a conductivity detector (calibrated against 99.99% CO<sub>2</sub>) to determine surface carbon of untreated chip samples. A mean value of 1.8 µg/g C was obtained for the surface carbon content of 10 samples.

Line 11: Calibrated with calcium carbonate.

All crucibles were heated for 30 minutes at 1350°C.

Line 12: Calibrated with sodium carbonate.

The external surface of the disc sample and the punch were carefully cleaned before punching. The hot crucible technique was used as in Reference (3) in the Explanatory Note.

Line 13: Calibrated with calcium carbonate.

The samples were surface ground on an alumina bett before punching. The punched samples were washed in acetone and dried in air. All crucibles were preheated at 1000°C for 2 hours.

## ECRM 097-1 (D) - SUPPLEMENTARY CERTIFICATE RE CARSON

# Explanatory Note re Certification of Carbon Content

The initial determinations of carbon in this material were carried out on the bulk chip sample prepared in 1984. The laboratory mean values obtained covered a wide range from 1 to 20 µg/g and hence it was not possible to certify the carbon content.

During the succeeding 14 year period much research has been carried out on the accurate determination of carbon in low carbon steels and irons. This work has revealed that freshly machined surfaces of these materials rapidly adsorb microgram amounts of carbon from the atmosphere and that the amount adsorbed is directly proportional to the surface area of the sample (1), (2) and (3).

During the last 10 to 15 years several instruments (some commercially available) have been developed for this determination and, in response to a demand for an iron base CRM certified for carbon at <5 µg/g, it was decided to carry out a project to certify the C content of ECRM 097-1. In order to reduce the surface area, it was decided to use only solid samples, cut or punched from 3mm thick discs of the 38mm dia. material (originally prepared for the production of discs for Optical Emission and X-Ray Fluorescence Spectrometry).

A number of laboratories, known to have experience in this determination, were invited to participate in this project. To minimise surface contamination the laboratories were asked to cut or punch samples from the disc material (after filling the surface) immediately before combustion, without any further treatment, other than washing in an organic solvent to remove any possible contamination during the cutting or punching process.

Some laboratories analysed both cut and punched samples, lower values were generally found on cut samples, which has been attributed to the exclusion of contamination in cracks formed round the edges of punched samples (3).

A VDEh Working Group has recommended a procedure for removing surface contamination from the sample by etching the surface with  $HF/H_2O_2$  (2) and as indicated in the Table of Mean Values this pretreatment was used by some of the laboratories.

An ISO Technical Committee Working Group has developed a Draft International Standard Method for this determination in which any engage contamination is removed by pre-heating the test portion at 410°C ± 10°C for 5 - 10 minutes immediately before combustion.

As pre-heating can only readily be carried out in a resistance furnace, some laboratories, using HE induction furnaces, have

As pre-heating can only readily be carried out in a resistance furnace, some laboratories, using HF induction furnaces, have found that pre-heating the crucible (with a pure copper flux) and placing the test portion in a hot crucible also removes surface contamination (3).

Some laboratories were able to monitor the evolution of CO<sub>2</sub> against time and found that there was an initial evolution after the first 10 seconds and the remainder within a further 70 seconds. The initial evolution has been regarded as being due to surface contamination of the sample.

The certified values for carbon given on this Certificate exclude surface contamination (or only include internal carbon) with the exception of the value on line 1 (see "Additional Information supplied by the participating laboratories").

#### References:

- (1) A Modified Combustion Method for the Determination of the Surface and Bulk Carbon Contents of High-Purity Metals. K. GRETZINGER, E. GRALLATH & G. TÖLG - Analytica Chimica Acta 193 (1987): 1-18.
- (2) Determination of Carbon in High-Purity Iron by a Modified Combustion Method.

  J-S. CHEN, U. BARTH and E. GRALLATH Fresenius Z Anal. Chem (1989) 334: 154-157.
- (3) Improvements in the Combustion Method for the Determination of Low Carbon Contents in Steel. W. GRUNER & E. GRALLATH Steel Research 66 (1995) No. 11: 455-457.

The Determination of C, S, O, N and H in High Purity Base Metals S. LAWRENZ - LECO Corporation, Michigan, USA.

- (5) Determination of Carbon in Steels and in Pure Iron in the range of 1 100 μg/g with Primary Calibration.
   D. FRANK & G. STAATS (1987) Fresenius Z Anal Chem 327: 456.
- (6) Unalloyed steel Determination of low carbon content Part 2: Infrared absorbtion method after combustion in an induction furnace (with pre-heating) ISQ/DIS 15349-2. See also ISQ/DTR 15349-1 and 15349-3.

### **FURTHER INFORMATION**

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