

# ECIIS

## 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. 197-1 Low Alloy Steel

EN Code 20MoCr4

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

Line No	C	Si	Mn	P	S	Cr	Mo	Ni	Al <sub>tot</sub>	As	Co	Cu	N
1	-	0.2623	0.7754	-	0.0211	-	0.3886	0.1434	0.0272	-	-	0.1451	0.0108
2	0.2120	0.2642	0.7805	0.0066	0.0213	0.4429	0.3949	0.1434	0.0289	-	0.0129	0.1463	0.0110
3	0.2148	0.2643	0.7819	0.0069	0.0218	0.4431	0.3961	0.1440	0.0294	0.0073	0.0130	0.1487	0.0110
4	0.2171	0.2688	0.7835	0.0070	0.0221	0.4455	0.3981	0.1458	0.0299	0.0076	0.0131	0.1498	0.0111
5	0.2175	0.2690	0.7868	0.0071	0.0228	0.4480	0.3990	0.1464	0.0301	0.0077	0.0133	0.1516	0.0111
6	0.2176	0.2695	0.7922	0.0072	0.0230	0.4484	0.3999	0.1467	0.0304	0.0077	0.0133	0.1520	0.0114
7	0.2182	0.2711	0.7925	0.0073	0.0231	0.4497	0.4006	0.1470	0.0304	0.0082	0.0133	0.1520	0.0114
8	0.2188	0.2718	0.7931	0.0074	0.0234	0.4500	0.4013	0.1480	0.0307	0.0082	0.0135	0.1526	0.0114
9	0.2189	0.2736	0.7936	0.0074	0.0237	0.4503	0.4015	0.1488	0.0317	0.0083	0.0136	0.1528	0.0115
10	0.2197	0.2752	0.7960	0.0074	0.0237	0.4506	0.4019	0.1489	0.0324	0.0084	0.0136	0.1548	0.0115
11	0.2200	0.2803	0.7963	0.0074	0.0240	0.4508	0.4032	0.1491	0.0328	0.0086	0.0136	0.1548	0.0117
12	0.2206	0.2829	0.7975	0.0074	0.0242	0.4517	0.4058	-	0.0344	0.0087	0.0136	0.1552	0.0117
13	0.2208	0.2830	0.8027	0.0077	0.0243	0.4561	0.4087	0.1515	0.0346	0.0088	0.0137	0.1556	0.0119
14	0.2212	0.2833	0.8121	0.0078	0.0249	0.4613	0.4121	0.1532	0.0349	0.0089	0.0139	0.1568	0.0120
15	0.2218	0.2896	-	0.0082	0.0252	0.4620	0.4152	0.1574	-	0.0092	0.0140	0.1574	0.0123
16	-	0.2975	-	-	-	-	-	-	-	0.0092	0.0143	-	-
<b>M<sub>M</sub></b>	<b>0.2185</b>	<b>0.2754</b>	<b>0.7917</b>	<b>0.0073</b>	<b>0.0232</b>	<b>0.4507</b>	<b>0.4018</b>	<b>0.1481</b>	<b>0.0313</b>	<b>0.0083</b>	<b>0.0135</b>	<b>0.1524</b>	<b>0.0114</b>
<b>S<sub>M</sub></b>	0.0027	0.0099	0.0096	0.0004	0.0012	0.0057	0.0067	0.0039	0.0023	0.0006	0.0004	0.0036	0.0004
<b>S<sub>w</sub></b>	0.0012	0.0034	0.0029	0.0002	0.0004	0.0023	0.0022	0.0012	0.0005	0.0003	0.0003	0.0012	0.0002

Line No	Sn	Ti	V
1	0.0084	0.0004	0.0025
2	0.0087	0.0004	0.0034
3	0.0089	0.0004	0.0038
4	0.0093	0.0004	0.0043
5	0.0093	0.0005	0.0045
6	0.0094	0.0005	0.0047
7	0.0094	0.0005	0.0052
8	0.0097	0.0005	0.0055
9	0.0098	0.0006	0.0056
10	0.0100	-	0.0056
11	0.0102	0.0008	0.0056
12	0.0105	-	0.0058
13	0.0106	-	0.0059
14	0.0109	-	0.0060
15	0.0109	-	-
<b>M<sub>M</sub></b>	<b>0.0097</b>	<b>0.0005</b>	
<b>S<sub>M</sub></b>	0.0007	0.0001	
<b>S<sub>w</sub></b>	0.0004	0.0001	

Additional values for information: Bi - 0.1 µg/g; Sb - 18 µg/g; Pb - 3 µg/g; (one lab)  
Al<sub>acid soluble</sub>: 0.0244 µg/g; 0.0297 µg/g (two labs)

M<sub>M</sub>: Mean of the intralaboratory means

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

S<sub>w</sub>: Intralaboratory standard deviation

S<sub>b</sub>: Interlaboratory standard deviation

$$S_b = \sqrt{S_M^2 - S_w^2 / 4}$$

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 the Grubbs Test.

Values given in italics are for information only.

#### CERTIFIED VALUES

	C	Si	Mn	P	S	Cr	Mo	Ni
<b>Mass content in %</b>	<b>0.219</b>	<b>0.275</b>	<b>0.792</b>	<b>0.0073</b>	<b>0.0232</b>	<b>0.451</b>	<b>0.402</b>	<b>0.148</b>
C(95%)	0.002	0.005	0.006	0.0003	0.0007	0.003	0.004	0.003

	Al <sub>tot</sub>	As	Co	Cu	N	Sn	Ti
<b>Mass content in %</b>	<b>0.0313</b>	<b>0.0083</b>	<b>0.0135</b>	<b>0.152</b>	<b>0.0114</b>	<b>0.0097</b>	<b>0.0005</b>
C(95%)	0.0014	0.0004	0.0003	0.002	0.0003	0.0005	0.0001

The half-width confidence interval C(95%)= t<sub>(n-1)</sub> × S<sub>M</sub> / √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.

This reference material was prepared in accordance with the recommendations set out in ISO Guides 30 – 35. This reference material was prepared and issued by:

**Corrosion and Metals Research Institute**  
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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 organisations.  
(France-IRSID/CTIF; Germany-Iron and Steel CRM Working Group: VDEh, BAM & MPI für Eisenforschung; UK-BAS Ltd; Nordic Countries-Nordic CRM Working Group)



## METHODS USED

Element	Line number	Analytical methods
C	2, 3, 4, 5, 6, 8, 9, 11, 12, 13, 14, 15 7 10	Combustion - infrared absorption Coulometric titration Non-aqueous titration after absorption in organic solvent
Si	1, 5, 6, 8, 14, 16 2, 3, 4, 7, 10, 11, 12, 15 9 13	Gravimetry, dehydration with perchloric acid ICP-OES Gravimetry, dehydration with nitrosulphuric acid MAS, molybdenum blue, without extraction
Mn	1, 2, 12 3, 4, 5, 7, 8, 9, 11, 13, 14 6, 10	MAS, periodate oxidation ICP-OES FAAS
P	2, 3, 4, 5, 9, 10, 15 6, 7, 8 11 12, 13 14	ICP-OES MAS, phosphovanadomolybdate, extraction MAS, molybdenum blue, extraction MAS, molybdenum blue, without extraction MAS, phosphovanamolybdate, without extraction
S	1, 2, 3, 4, 5, 6, 7, 8, 10, 11, 12, 13, 14, 15 9	Combustion – infrared absorption Gravimetry as BaSO <sub>4</sub> without separation
Cr	2, 14 3, 4, 12 5, 6, 7, 8, 9, 10, 11, 13, 15	Titration with Fe (II), oxidation with persulphate FAAS ICP-OES
Mo	1, 2, 3, 5, 6, 8, 9, 10, 11, 14 4, 12 13, 15 7	ICP-OES FAAS MAS, thiocyanate in presence of Sn (II), extraction MAS, thiocyanate in presence of ascorbic acid, extraction
Ni	1, 7, 8 2, 3, 5, 6, 9, 10, 11, 13, 14, 15 4	FAAS ICP-OES MAS, dimethylglyoxime, extraction
Al(tot)	1, 5, 6, 7, 12, 13, 14 2, 3, 8, 10, 11 4 9	ICP-OES FAAS MAS, eriochrome cyanine, sodium hydroxide separation of iron ICP-MS
As	3 4, 5, 12, 16 6, 8, 10, 14 7, 9, 13 11 15	MAS, molybdenum blue, separation as arsine ICP-OES ETAAS ICP-MS MAS, diethyldithiocarbamate, separation as arsine Titration with Ce(IV), precipitation of elemental As
Co	2, 5, 7, 8, 9, 11, 12, 13, 15 3, 16 4 6 10 14	ICP-OES ICP-MS MAS, Nitroso R salt Electrogravimetry FAAS MAS, 2-nitroso-1-naphthol, without extraction
Cu	1, 7, 8 2, 3, 4, 5, 9, 10, 11, 12, 13, 14, 15 6	FAAS ICP-OES MAS, diethyldithiocarbamate, extraction
N	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14 15	Thermal conductivity, decomposition in graphite crucible Acidimetric titration after distillation, visual end point
Sn	1, 4, 12 2, 3, 6, 7, 8, 10 5, 9, 15 11 13, 14	ICP-MS ICP-OES ETAAS AAS, hydride generation FAAS
Ti	1, 2, 3, 4, 6, 7, 8, 11 5, 9	ICP-OES ICP-MS
V	1, 2, 8, 9, 10, 11, 12, 13, 14 3, 4 5 6, 7	ICP-OES FAAS MAS, N-benzoylphenylhydroxylamine, extraction ICP-MS
Bi		ICP-MS
Sb		ICP-MS
Pb		ICP-MS
Al <sub>acid soluble</sub>		FAAS

## Abbreviations:

FAAS	Flame Atomic Absorption Spectrometry
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ETAAS	Electrothermal Atomic Absorption Spectrometry
MAS	Molecular Absorption Spectrometry

## **DESCRIPTION OF THE SAMPLE**

The ECRM 197-1 is available in the form of milling chips in bottles containing 100 g. It is also available as 38 mm diameter discs 25 mm thick. The chips were passed through a 2000 µm aperture sieve and further sieving was carried out to exclude chips passing through a 250 µm aperture sieve.

## **INTENDED USE & STABILITY**

The chip sample, ECRM 197-1, 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 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 197-1, is intended for establishing and checking the calibration of instruments, such as Optical Emission Spectrometers 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 in order 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 ECRM 197-1 has been established in accordance with the 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, 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, 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 produced by the Korrosions- och Metallforskningsinstitutet AB may be obtained from the address given below.

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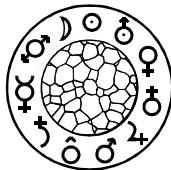
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