

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. **274-1** VANADIUM STEEL

LABORATORY MEANS (4 Values) mass content in %

Line No	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	N	V	W	O ^(*)	Al _{tot}
1	1.5450	1.0273	0.3848	0.0134	0.0086	7.9580	-	0.0670	-	0.0724	-	0.0072	0.0018	0.0005
2	1.5499	-	0.3881	0.0140	0.0087	7.9950	-	0.0735	0.0267	0.0744	-	0.0073	0.0021	0.0015
3	1.5500	1.0390	0.3887	0.0144	0.0087	8.0012	1.3930	0.0740	0.0267	0.0753	3.9578	0.0075	0.0021	0.0018
4	1.5547	1.0467	0.3900	0.0144	0.0088	8.0105	1.4177	0.0742	0.0267	0.0760	3.9625	0.0078	0.0021	0.0024
5	1.5580	1.0496	0.3905	0.0145	0.0093	8.0134	1.4293	0.0750	0.0271	0.0762	3.9666	0.0081	0.0022	0.0025
6	1.5586	1.0526	0.3907	0.0145	0.0094	8.0152	1.4320	0.0764	0.0278	0.0764	3.9703	0.0084	0.0023	0.0026
7	1.5600	1.0537	0.3920	0.0146	0.0095	8.0153	1.4480	0.0775	0.0279	0.0770	3.9879	0.0084	0.0026	0.0026
8	1.5620	1.0558	0.3958	0.0146	0.0095	8.0381	1.4487	0.0777	0.0282	0.0770	3.9968	0.0086	0.0026	0.0029
9	1.5645	1.0591	0.3972	0.0147	0.0098	8.0461	1.4553	0.0779	0.0282	0.0772	3.9973	0.0086	0.0026	0.0030
10	1.5660	1.0632	0.3982	0.0147	0.0098	8.0491	1.4586	0.0781	0.0284	0.0772	3.9975	0.0088	0.0028	0.0037
11	1.5667	1.0649	0.3991	0.0150	0.0098	8.0591	1.4595	0.0783	0.0284	0.0772	4.0024	0.0090	0.0029	0.0039
12	1.5717	1.0685	0.4017	0.0152	0.0099	8.0734	1.4650	0.0788	0.0285	0.0777	4.0042	0.0093	0.0030	-
13	1.5740	1.0692	0.4024	0.0154	0.0099	8.0840	1.4679	0.0795	0.0285	0.0780	4.0060	0.0100	0.0030	-
14	1.5758	1.0724	0.4030	0.0159	0.0104	8.0900	1.4737	0.0804	0.0285	0.0794	4.0146	0.0107	0.0030	-
15	1.5760	1.0771	0.4070	0.0165	0.0109	8.0922	1.4858	0.0805	0.0288	0.0795	4.0197	0.0112	0.0032	-
16	1.5761	-	0.4078	-	0.0110	-	1.4916	-	0.0290	0.0800	4.0413	-	0.0032	-
17			0.4097				1.5002	0.0868	0.0296		4.0461			
18											4.0623			
19											4.0662			
20											4.0800			
M _M	1.5630	1.0568	0.3970	0.0148	0.0096	8.0360	1.4551	0.0770	0.0281	0.0769	4.0100	0.0087		
S _M	0.0100	0.0139	0.0075	0.0008	0.0007	0.0393	0.0287	0.0042	0.0009	0.0019	0.0366	0.0012		
S _W	0.0060	0.0059	0.0035	0.0004	0.0003	0.0224	0.0085	0.0013	0.0003	0.0008	0.0185	0.0003		

Line No	As	Co	Pb	Sn	Ti	Sb
1	0.0013	0.0212	0.000060	0.00081	0.0006	0.000185
2	0.0013	0.0213	0.000065	0.00093	0.0012	0.000190
3	0.0014	0.0226	0.000068	0.00095	0.0014	0.000200
4		0.0266		0.00150		

Additional value for information: B ~ 5 µg/g

Values given in italic type are for information only

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

$$s_b = \sqrt{s_M^2 - s_w^2 / 4}$$

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 refer to solid samples.

CERTIFIED VALUES

	C	Si	Mn	P	S	Cr	Mo	Ni	Cu
M _M	1.563	1.057	0.397	0.0148	0.0096	8.036	1.455	0.077	0.0281
C(95%)	0.006	0.008	0.004	0.0005	0.0004	0.022	0.016	0.002	0.0005

	N	V	W
M _M	0.0769	4.010	0.0087
C(95%)	0.0010	0.018	0.0007

The half-width confidence interval C(95%)=t_(n-1) × s_M / √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 in accordance with the recommendations set out in ISO Guides 30 – 35 and issued by:

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P.O. Box 55970, SE 102 16, Stockholm, Sweden

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 (NCRMWG))



Certified Institute



Revised June 2009

METHODS USED

Element	Line number	Analytical methods
C	1, 3, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16	Combustion - infrared absorption
	2	Coulometric titration
	4	Non-aqueous titration after absorption in organic solvent
	1, 8, 10, 13,	ICP - OES
Si	3, 4, 5, 6, 7, 9, 11, 12, 15	Gravimetry, dehydration with perchloric acid
	14	Gravimetry, dehydration with hydrochloric acid
	1, 3, 4, 6, 7, 8, 9, 10, 11, 14, 17	ICP - OES
Mn	2, 5, 13, 15	FAAS
	12, 16	MAS, periodate oxidation
	1, 3, 4, 8, 12, 14	ICP - OES
P	2, 9, 13	MAS, molybdenum blue, without extraction
	5	Acidimetric titration of ammonium phosphomolybdate
	6, 7, 10, 15	MAS, phosphovanadomolybdate, extraction
	11	MAS, molybdenum blue, extraction
S	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 13, 14, 15, 16	Combustion - infrared absorption
	11	Gravimetry as BaSO ₄ without separation
Cr	1, 5, 6, 7, 8, 9, 10, 12	Titration with Fe (II), oxidation with persulphate
	2	FAAS
	3, 4, 13, 14, 15	ICP - OES
	11	Titration with Fe (II), oxidation with perchloric acid
Mo	3, 4, 6, 7, 8, 9, 13, 17	ICP - OES
	5, 11, 15, 16	FAAS
	10, 12, 14	MAS, thiocyanate in presence of Sn (II), extraction
Ni	1, 5, 6, 10, 13	FAAS
	2, 3, 4, 8, 9, 11, 12, 14, 15, 17	ICP - OES
	7	MAS, dimethylglyoxime, extraction
Cu	2, 3, 5, 6, 8, 10, 12, 13, 16	ICP - OES
	4, 7, 9, 11, 14, 15, 17	FAAS
N	1	Acidimetric titration after distillation, visual end point
	2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16	Thermal conductivity, decomposition in graphite crucible
V	3, 4, 9, 12, 13, 14, 15, 20	Titration with Fe (II), oxidation with Mn (VII)
	5, 6, 8, 11, 17, 18, 19	ICP - OES
	7, 10, 16	FAAS
W	1	MAS, thiocyanate formed in a strongly acid reducing medium
	2, 4, 8, 10, 11, 14, 15	ICP - OES
	3, 5, 6, 9, 12, 13	ICP - MS
	7	NAA
O	1, 2, 3, 4, 5, 6, 7, 8, 9, 11, 12, 13, 14, 15, 16	Infrared absorption measurement reduction fusion under helium
	10	Thermal conductivity, reduction fusion under nitrogen
Al_(tot)	1	ETAAS
	2, 3, 5, 8, 10	FAAS
	4, 6, 7, 9, 11	ICP - OES
As	1, 2	ICP - MS
	3	NAA
Co	1, 3, 4	ICP - MS
	2	ICP - OES
Pb	1, 2, 3	ICP - MS
Sn	1, 2, 3	ICP - MS
	4	ICP - OES
Ti	1	ICP - OES
	2,3	ICP - MS
Sb	1, 2, 3	ICP - MS

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
NAA	Neutron Activation Analysis

DESCRIPTION OF THE SAMPLE

The ECRM 274 -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 274-1, 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 274-1, 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.

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 CEN Report CR 10317 and ECISS Information Circular No. 5, 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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