



REPLICAST LTD.

and

BUREAU OF ANALYSED SAMPLES LTD.



CERTIFICATE OF ANALYSIS

SPECTROSCOPIC CERTIFIED REFERENCE MATERIAL

SCRM No. 658/12

LOW PHOSPHORUS ENGINEERING IRON

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, issued by the Bureau of Analysed Samples Ltd., Newham Hall, Middlesbrough, England.

CO-OPERATING ANALYSTS

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 Ridsdale & Co Ltd., Middlesbrough.
 Element Materials Technology, Rotherham.
 AMG Analytical Services, Rotherham.

ANALYSES

Mean of 4 values – mass content in %.

Analyst No.	C	Si	Mn	P	S
1	3.3400	2.0360	0.5685	0.2423	0.0764
2	3.3265	2.0392	0.5350	0.2429	0.0758
3	3.3207	2.0335	0.5608	0.2500	0.0793
4	3.3745	2.0175	0.5600	0.2465	0.0748
5	3.3193	2.0400	0.5506	0.2335	0.0775
M_M	3.3362	2.0332	0.5550	0.2430	0.0768
SM	0.0229	0.0092	0.0129	0.0062	0.0018
SW	0.0081	0.0075	0.0029	0.0033	0.0008

M_M: Mean of the laboratory mean values. **SM**: standard deviation of the laboratory mean values. **SW**: average within laboratory standard deviation.

Additional Information: Analysts Nos. 3 and 4 both determined Cr and Ti by ICP-OES and each reported a value of 0.01% for Cr and 0.02% for Ti.

Lab 3 also determined the following elements by ICP-OES: Mo, Al, Sn and Mg (all <0.001%), Ni (0.004%), Co (0.001%), Cu (0.004%), V (0.006%) and Ce (0.003%)

CERTIFIED VALUES (C_v)

mass content in %

	C	Si	Mn	P	S
C_v	3.336	2.033	0.555	0.243	0.0768
C(95%)	0.029	0.012	0.016	0.008	0.0022

The half width confidence interval, C(95%), is an expression of the uncertainty of the certified value.

$C(95\%) = \frac{t \times SM}{\sqrt{n}}$ where “t” is the appropriate two sided Student’s t value at the 95% confidence level for “n” acceptable mean values.

For further information regarding the confidence interval for the certified value see ISO Guide 35.

NB: Although widely accepted within the industry “mass content in %” is neither an SI nor an IUPAC supported quantity. Multiplication of the certified value (C_v) by 10⁴ will yield the value in µg/g.

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NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 4 and 5 determined carbon using high frequency combustion and infrared absorption. Analysts Nos. 2 and 3 used gravimetric methods after combustion in a stream of oxygen, Analyst No. 3 using the standard method BS 6200.3.8.3:1990.

SILICON

Analysts Nos. 1, 4 and 5 determined silicon using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The remaining Analysts determined silicon gravimetrically, dehydrating the silica using perchloric acid, according to BS 6200: 3.26.1: 1995.

MANGANESE

Analyst No. 2 determined manganese by Flame Atomic Absorption Spectrometry (FAAS) whilst No. 3 used a photometric method, oxidising with periodate. The remaining Analysts used ICP-OES.

PHOSPHORUS

Analysts Nos. 2 and 3 determined phosphorus photometrically as phosphovanadomolybdate, extracting the complex with 4-methyl pentan-2-one. The remaining analysts used ICP-OES

SULPHUR

Analysts Nos. 1, 4 and 5 determined sulphur by high frequency combustion and infrared absorption. Analyst No.2 used an acid base titration following combustion in a stream of oxygen whilst No. 3 used ICP-OES.

DESCRIPTION OF SAMPLE

The material for this SCRM was prepared by Replicast Ltd. at Sheffield, U.K using a special method of casting known to provide material of uniform composition in a form suitable for use as a Certified Reference Material (CRM) for optical emission spectrometric analysis. Blocks from this cast have been shown, by statistically designed procedures, to provide reproducible results using optical emission spectrometry.

SCRM 658/12 is sold in the form of chill cast rectangular blocks, each approximately 48mm x 42mm x 12mm thick with a single chilled working face. Spectroscopic reproducibility has been shown to be reliable to a depth of 5mm below the original surface of this block. Sparking must be made on the fully ground surface only and the sample should be discarded when this face has been ground back as far as the small shoulder around the edge of the sample.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of SCRM 658/12 as defined in BS EN ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with BS EN ISO 17034 and ISO Guide 35.

INTENDED USE

SCRM 658/12 is primarily intended for the construction of calibration curves or for Quality Control purposes. It is one of a series of such SCRMs (SCRMs 656 – 660 et seq) which may be used together. Users should be aware that, when using optical emission spectrometers, a minimum of three sparks, made on separate, clean locations across the face of the block, should be obtained in order to establish a reliable mean value.

STABILITY

This SCRM will remain stable provided that the blocks are stored in a dry atmosphere.

TRACEABILITY

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above. The analytical sample was prepared by taking turnings from several blocks which were then mixed and subdivided. Each analyst received a representative sample of the bulk material and the Certified Values accurately represent the chemical composition of the SCRM.

Most of the analytical methods used in the characterisation of this SCRM were either international or national standard methods or methods which are technically equivalent. All laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of SCRM 658/12 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's t value at the 95% confidence level for the number of results. Homogeneity has been assessed in accordance with ASTM E826 – 85 and found to be acceptable. It has not, therefore, been included in the calculated measurement uncertainty. The stability of this SCRM and the transportation of the blocks also make negligible contributions to the overall uncertainty of the certified values.

COMMUTABILITY

When using optical emission spectrometers it has been established that materials of similar composition from different sources may respond differently. The user should be aware that, although similar, the metallurgical history of this SCRM may not be identical to that of other SCRMs in this series and may not accurately reflect the metallurgical history of the user's own materials.

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.

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