Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

Indian Standard

SPECTROMETRIC ANALYSIS OF CAST IRON
BY DIRECT READING OPTICAL EMISSION VACUUM
SPECTROMETER—POINT TO PLANE TECHNIQUE

ICS 77.080.10

© BIS 2003

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 12308 ‘Methods for chemical analysis of cast iron and pig iron’ in parts covers the chemical analysis of various constituents in cast iron and pig iron. Since instrumental techniques are now widely adopted for rapid and accurate analysis a need was felt to prepare this standard. In this standard the condensed spark method is specified for determination of alloying elements in cast iron with the help of direct reading optical emission spectrometer having automatic print out typewriter/strip chart recorder.

The methods specified are intended to be broad guidelines only, and the complete specifications for all electrical and electronic characteristics will depend on the various instruments commercially available and nature of analytical programme.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (revised)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
Indian Standard

SPECTROMETRIC ANALYSIS OF CAST IRON
BY DIRECT READING OPTICAL EMISSION VACUUM
SPECTROMETER—POINT TO PLANE TECHNIQUE

1 SCOPE
This standard prescribes the spectrometric method (point to plane technique) of analysis with the help of direct reading optical emission spectrometer for the determination of carbon, manganese, phosphorus, sulphur, silicon, copper, chromium, nickel, aluminium, molybdenum, titanium, magnesium and vanadium in cast iron.

2 SAMPLING

2.1 Sample Mould
Capable of producing casting that are homogeneous and free from voids and porosity. The mould made up of copper has been found to produce acceptable sample. The prepared face should be free from precipitated graphite which always interferes with the sparking and proper integration does not occur. So the sample is poured in a coin of minimum thickness of 5 mm mould made of copper. The bottom surface which is chilled due to direct contact with the mould is prepared for sparking. Chilling gives white structure to the iron where all the carbon is held in combined form. The process of rapid chilling maybe enhanced by enclosing the mould in a small water jacket connected with inflow and outflow of cold water supply. The cast iron sample is taken out for further processing. Alternately sampling can be done using in lolly pop sampler without aluminium addition for killing.

2.2 Preparation of the Sample
Cast iron sample as casted in 2.1 shall be ground in a surface grinder fitted with magnetic/mechanical chuck. As the surface gets heated quickly, wet grinding method is preferred. A smooth and clean surface is exposed and finally the coolant or any adhering material is removed with one or two sweeps of dry grinding. Finally, to achieve uniform surface, the sample is polished on a horizontal polishing machine using 60-80 grit paper and a magnetic holder. Overheating during grinding and polishing, which may cause oxidation, shall be avoided.

NOTE — At least two samples should be cast for each heat.

2.3 Sample Size and Shape
To facilitate handling the main dimensions may be in the range of:

a) Diameter — 20-40 mm, and
b) Thickness — 5 mm, Min.

However, the parameters may vary depending upon different makes of direct-reader instruments. In practice, the prepared face should be large enough to accommodate 2-3 sparks without the need for refacing. The top surface should be levelled properly to allow the sample to be clamped in the required position.

3 APPARATUS

3.1 Spectrometer — Any standard vacuum optical emission spectrometer with a minimum reciprocal dispersion of 0.5 to 0.6 nm/mm shall be suitable for use. The final output can be in a digital mode print or microprocessed.

3.2 Source Unit — A source unit capable of producing a triggered capacitor discharge is suitable. Typical source parameters are given below:

- Capacitance — 10 microfarad
- Inductance — 310 microhenries
- Resistance — 0.1 ohm

3.3 Spark Stand — Suitable for mounting in optical alignment a flat surface of the specimen in opposite to a silver/thoriated tungsten electrode.

NOTES
1. Clean the spark stand, lens or quartz window to minimize the transmission losses. Frequency of cleaning is to be established by individual laboratories.
2. Reshape the silver/tungsten electrode when necessary and clean the electrode tip with a cleaning brush after every spark.

3.4 Argon Flushing System — System consists of argon cylinders, a two stage leak proof regulator, a gas flow meter and connecting copper/stainless steel tubing of 4-6 mm diameter to provide a controlled inert atmosphere during sparking. Total impurities in argon shall not exceed 50 ppm.

3.5 Temperature Control — The whole instrument, that is source unit, spectrometer and the printer should be housed in a fully air conditioned room maintained at a temperature and humidity specified by the manufacturer. The temperature should be maintained to reduce shift in spectral lines due to temperature variation. The room should be free from vibrations, dust and oil vapsours arising from the outlets of vacuum pump.

3.6 Vacuum System — To reduce the absorption of spectral radiation in the ultra-violet region and avoid
oxidation, it is necessary to remove the air inside the spectrometer through which the radiation is passing. A suitable vacuum system as recommended by the equipment manufacturer may be used for this purpose.

NOTE — In case of spectrometers with multiple spectrometric chambers, a nitrogen filled chamber must be provided for the wavelengths in the UV range.

3.7 Spectral Lines and Concentrations — Typical spectral lines along with the concentration ranges are given in Table 1 for guidance. Use suitable iron line is used as the internal standard.

3.8 Spark and Measurement of Spectra — Programme the spectrometer to accommodate the internal standard lines and one of the analytical lines for each element listed in Table 1. Place the prepared surface of the sample on the spark stand and excite twice at different spots. A suitable gap is to be maintained between two electrodes by gauge. Take the average measured intensity of the two sparks. A good spark is indicated by the dark round ring around the sparked area. Results on sparks without a dark ring should be rejected. Measure the intensity, at the analytical line recommended for each element, compare it with that of similar standard and compute the concentration of the element.

4 CALIBRATION AND STANDARDIZATION

Standardization is carried out using standard sample and following the procedure specified in 3.8.

NOTE — Due to metallurgical difference between the primary standards and the production sample it is preferable to check up the standardization using secondary standards of similar chemical composition and of the same metallurgical history as that of the sample and to make the corrections, if necessary.

Table 1 Spectral Lines and Concentration Ranges
(Clause 3.7 and 3.8)

<table>
<thead>
<tr>
<th>SI No.</th>
<th>Element</th>
<th>Wave Length (nm)</th>
<th>Concentration Range (Percentage by Mass)</th>
</tr>
</thead>
<tbody>
<tr>
<td>i)</td>
<td>Fe</td>
<td>271.4</td>
<td>0.003-4.0</td>
</tr>
<tr>
<td>ii)</td>
<td>C</td>
<td>193.09</td>
<td>0.002-0.5</td>
</tr>
<tr>
<td>iii)</td>
<td>Mn</td>
<td>348.29</td>
<td>0.21-18.0</td>
</tr>
<tr>
<td>iv)</td>
<td>P</td>
<td>178.2</td>
<td>0.002-0.5</td>
</tr>
<tr>
<td>v)</td>
<td>S</td>
<td>180.7</td>
<td>0.002-0.2</td>
</tr>
<tr>
<td>vi)</td>
<td>Si</td>
<td>212.4</td>
<td>0.1-5.0</td>
</tr>
<tr>
<td>vii)</td>
<td>Si</td>
<td>251.6</td>
<td>0.002-1.0</td>
</tr>
<tr>
<td>viii)</td>
<td>Cu</td>
<td>327.4</td>
<td>0.01-0.5</td>
</tr>
<tr>
<td>ix)</td>
<td>Cr</td>
<td>267.7</td>
<td>0.003-1.5</td>
</tr>
<tr>
<td>x)</td>
<td>Ni</td>
<td>243.48</td>
<td>1.0-15.0</td>
</tr>
<tr>
<td>xi)</td>
<td>Al</td>
<td>394.4</td>
<td>0.001-1.5</td>
</tr>
<tr>
<td>xii)</td>
<td>Mo</td>
<td>386.41</td>
<td>0.1-0.6</td>
</tr>
<tr>
<td>xiii)</td>
<td>V</td>
<td>311.02</td>
<td>0.01-3.0</td>
</tr>
</tbody>
</table>
Bureau of Indian Standards

BIS is a statutory institution established under the Bureau of Indian Standards Act, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publication), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. MTD 2 (2530).

Amendments Issued Since Publication

<table>
<thead>
<tr>
<th>Amend No.</th>
<th>Date of Issue</th>
<th>Text Affected</th>
</tr>
</thead>
</table>

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephone: 2323 0131, 2323 3375, 2323 9402

Regional Offices:
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002
Telephone: 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi
KOLKATA 700054
Telephone: 2337 8499, 2337 8561

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022
Telephone: 2337 8626, 2337 9120

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113
Telephone: 2254 1216, 2254 1442

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093
Telephone: 2832 9295, 2832 7858

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD.
GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR.
NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Printed at Simco Printing Press, Delhi