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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”

SP 71 (2012): Compendium of Method of Chemical Analysis of Steels [MTD 4: Wrought Steel Products]
COMПENDIUM OF METHOD OF CHEMICAL ANALYSIS OF STEELS

ICS 77.080.01
FOREWORD

This Special Publication was adopted by the Bureau of Indian Standards, after the draft finalized by the Wrought Steel Products Sectional Committee had been approved by the Metallurgical Engineering Division Council. The Committee in one of its meeting decided to adopt ISO/TR 9769 : 1990 ‘Steel and iron — Review of available methods of analysis’. This is an important publication and has been cross referred in number of other ISO Standards such as ISO 404 : 1992 ‘Steel and steel products — General technical delivery requirements’, etc. Bureau of Indian Standards has also not formulated any publication similar to this publication. In the light of the above, it has been decided to formulate a corresponding Special Publication to bridge the gap in the standardization.

For all the tests specified in this publication, the method as specified in relevant ISO Standard may also be followed as an alternate method.

While formulating this publication assistance has been derived from ISO/TR 9769.

The composition of the Committee responsible for formulation of this Special Publication is given in Annex A.
1 SCOPE

1.1 This special publication aims to facilitate reference to the available Indian Standard on test methods for the determination of required element(s) in steel(s).

1.2 In this publication, the scope and outline (principal) of each standard are stated.

2 SYNOPSIS OF METHOD

2.1 Carbon

2.1.1 IS 228 (Part 1) : 1987 ‘Methods for chemical analysis of steels: Part 1 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent) (third revision)’

Scope — This standard (Part 1) covers volumetric method for determination of carbon in the range 0.05 to 2.50 percent in plain carbon, low alloy and high alloys steels.

Outline of the method — The sample is burnt in a current of pure oxygen in presence of a suitable flux. Combustion of the sample in a stream of oxygen, thus converts all the carbon present to carbon dioxide. After removal of sulphurous gases by suitable absorbents, the carbon dioxide gas is collected in a specially jacketed burette along with excess of oxygen. The carbon dioxide is then absorbed in alkali. On passing the excess oxygen back to the burette, the contraction in volume is read against a scale, calibrated directly to the percentage of carbon.

2.1.2 IS 228 (Part 4) : 1987 ‘Methods for chemical analysis of steels: Part 4 Determination of total carbon by gravimetric method (for carbon > 0.1 percent) (third revision)’

Scope — This standard (Part 4) covers method for the determination of total carbon content of plain carbon, low alloy and high alloy steels of 0.1 percent and above by the gravimetric method.

Outline of the method — The sample is burnt in a current of pure oxygen in presence of a suitable flux. Combustion of the sample in a stream of oxygen, thus converts all the carbon present to carbon dioxide. After removal of sulphurous gases by suitable absorbents, the carbon dioxide gas is collected in a specially jacketed burette along with excess of oxygen. The carbon dioxide is then absorbed in alkali. On passing the excess oxygen back to the burette, the contraction in volume is read against a scale, calibrated directly to the percentage of carbon.

2.1.3 IS 228 (Part 14) : 1988 ‘Methods for chemical analysis of steels: Part 14 Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)’

Scope — This standard (Part 14) covers a method for determination of carbon in all types of steels and alloy steels in the range of 0.005 to 2.000 percent.

Outline of the method — The sample is burnt in a stream of oxygen in presence of a metal accelerator. The carbon dioxide formed is selectively adsorbed on the molecular sieve at a temperature and released by heating at 300ºC. The detector is a thermistor cell which senses the difference between thermal conductivity of the carrier gas (with helium specially extra-low carbon and oxygen in other cases) and that of the carrier gas containing carbon dioxide. The difference is proportional to carbon content of the sample.

2.1.4 IS 228 (Part 20) : 2003 ‘Methods for chemical analysis of steels: Part 20 Determination of carbon and sulphur by infra red absorption method (for carbon 0.005 to 2 percent and sulphur 0.001 to 0.35 percent)’

Scope — This standard (Part 20) describes the method for determination of carbon in the range from 0.005 to 2 percent and sulphur from 0.001 to 0.35 percent in steel.

Outline of the method — The sample is burnt with accelerator at high temperature in a flow of oxygen in a high frequency induction furnace. The oxygen flow serves both as oxidizing agent and as system carrier gas. Carbon in the ample is converted to carbon dioxide and sulphur to sulphur dioxide. These are carried by oxygen stream into the non-dispersive infrared absorption analyzer where these gases are measured quantitatively.

2.2 Manganese

2.2.1 IS 228 (Part 2) : 1987 ‘Methods for chemical analysis of steels: Part 2 Determination of manganese in plain-carbon and low alloy steels by arsenite method (third revision)’

Scope — This standard (Part 2) covers method for the determination of manganese in plain carbon and low alloy steels by arsenite method.

Outline of the method — Manganese is
oxidized, in presence of silver nitrate, to permanganic acid by ammonium persulphate and titrated with sodium arsenite solution.

2.2.2 IS 228 (Part 12) : 2001 ‘Methods for chemical analysis of steels: Part 12 Determination of manganese by periodate spectrophotometric method in plain carbon, low alloy and high alloy steels (for manganese 0.01 to 5.0 percent) (fourth revision)’

Scope — This standard (Part 12) describes the method for the determination of manganese in plain carbon, low alloy and high alloy steels in the range of 0.01 to 5.0 percent by periodate spectrophotometric method.

Outline of the method — After dissolution of the sample in sulphuric-phosphoric acid mixture and nitric acid, the solution is fumed with perchloric acid. Manganese is oxidized to permanganic acid by potassium periodate. Absorbance of the solution is measured at 545 nm.

2.3 Phosphorus

2.3.1 IS 228 (Part 3) : 1987 ‘Methods for chemical analysis of steels: Part 3 Determination of phosphorus by alkalimetric method (third revision)’

Scope — This standard (Part 3) covers method for determination of phosphorus content of plain carbon steel and alloy steels by alkalimetric method.

Outline of the method — Phosphorus is converted to orthophosphoric acid and precipitated as ammonium phosphomolybdate. The precipitate is dissolved in known excess of standard sodium hydroxide solution and the excess is titrated against standard nitric acid solution.

2.4 Nickel

2.4.1 IS 228 (Part 5) : 1987 ‘Methods for chemical analysis of steels: Part 5 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel > 0.1 percent) (third revision)’

Scope — This standard (Part 5) covers the method for determination of nickel content of plain carbon, low alloy and high alloy steels of 0.1 percent and above by the gravimetric method.

Outline of the method — After complexing interfering elements, nickel in the solution of the sample is precipitated as nickel dimethylglyoximate and weighed.

2.5 Chromium

2.5.1 IS 228 (Part 6) : 1987 ‘Methods for chemical analysis of steels: Part 6 Determination of chromium by persulphate oxidation method (for chromium > 0.1 percent) (third revision)’

Scope — This standard (Part 6) covers the persulphate oxidation method for determination of chromium content of low alloy and high alloy steels containing more than or equal to 0.1 percent chromium. This method is not applicable for steels containing tungsten.

Outline of the method — After dissolution of the sample in dilute sulphuric acid and phosphoric acid mixture and further treated with nitric acid, chromium, manganese and (vanadium if present) are oxidized by ammonium persulphate in presence of silver nitrate as catalyst. Permanganic acid is then destroyed by dilute hydrochloric acid. Chromium is reduced by ferrous ammonium sulphate and excess of ferrous ammonium sulphate is back titrated with standard potassium permanganate solution.

2.6 Molybdenum

2.6.1 IS 228 (Part 7) : 1990 ‘Methods for chemical analysis of steels: Part 7 Determination of molybdenum by alpha-benzoinoxime method in alloy steels (for molybdenum > 1 percent and not containing tungsten) (third revision)’

Scope — This standard (Part 7) covers the alphabenzoinoxime method for determination of molybdenum content in low alloy and high alloy steels containing molybdenum above 1 percent and no tungsten.

Outline of the method — Molybdenum is precipitated with alpha-benzoinoxime and the precipitate is ignited at 500-525°C and weighed as MoO₃.

2.6.2 IS 228 (Part 10) : 1989 ‘Methods for chemical analysis of steels: Part 10 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.50 percent) (third revision)’

Scope — This standard (Part 10) covers the method for the determination of molybdenum in the range 0.01 to 1.50 percent by thiocyanate (photometric) method in low and high alloy steels.

Outline of the method — Sample aliquot is
treated with thioicyanate to develop molybdenum and iron complexes and reduced with stannous chloride. Molybdenum complex is extracted with butyl acetate and measured at 470 nm.

2.7 Silicon

2.7.1 IS 228 (Part 8) : 1989 ‘Methods for chemical analysis of steels: Part 8 Determination of silicon by the gravimetric method (for silicon 0.05 to 5.00 percent) (third revision)’

Scope — This standard (Part 8) covers the gravimetric method for determination of silicon (0.05 to 5.00 percent) in plain carbon and alloy steels including high speed steels containing tungsten.

Outline of the method — After dissolution of the sample, silicic acid is dehydrated by fuming with sulphuric acid. The solution is filtered and silica is ignited, weighed and then volatilized with hydrofluoric acid. The residue is ignited and weighed; the loss in weight represent silica.

2.7.2 IS 228 (Part 11) : 1990 ‘Methods for chemical analysis of steels: Part 11 Determination of total silicon by reduced molybodosilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent) (third revision)’

Scope — This standard (Part 11) covers the spectro-photometric method for determination of total silicon in the range of 0.01 to 0.05 percent in carbon steels and low alloy steels.

Outline of the method — Silicon is converted to silicic acid and complexed with sodium molybdate which in turn is reduced to molybdenum blue. The absorbance of complex is measured at 810 nm.

2.8 Sulphur

2.8.1 IS 228 (Part 9) : 1989 ‘Methods for chemical analysis of steels: Part 9 Determination of sulphur by evolution method (for sulphur 0.01 to 0.25 percent) (third revision)’

Scope — This standard (Part 9) covers method for determination of sulphur in plain carbon and low alloy steel by evolution method.

NOTE — This method is not suitable for steel samples containing selenium.

Outline of the method — Sulphur is evolved as hydrogen sulphide and precipitated as cadmium sulphide in ammoniacal cadmium chloride solution. Solution is acidified and excess of potassium iodate-potassium iodide is added. Excesses of liberated iodine is titrated against sodium thiosulphate.

2.8.2 See also 2.1.4.

2.9 Arsenic

2.9.1 IS 228 (Part 13) : 1982 ‘Methods for chemical analysis of steels: Part 13 Determination of arsenic’

Scope — This standard (Part 13) prescribes the method for determination of arsenic in steel.

Outline of the method — Arsenic which in steel exists mainly as iron arsenide (FeAs₂) is converted into arsenic acid (H₃AsO₃) when the alloy is dissolved in a mixture of sulphuric acid and nitric acid. Arsenic acid is reduced quantitatively in acid medium by sodium hypophosphite to metallic arsenic and the metal is then oxidized by a measured excess of standard iodine solution to pentavalent arsenic. The excess iodine is back titrated with standard arsenious oxide solution.

2.10 Copper

2.10.1 IS 228 (Part 15) : 1992 ‘Methods for chemical analysis of steels: Part 15 Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent) (second revision)’

Scope — This standard (Part 15) describes the thiosulphate-iodide method for determination of copper in steel in range from 0.05 to 5 percent.

Outline of the method — The sample is dissolved in dilute sulphuric acid and copper is precipitated with sodium-thiosulphate. Precipitate is ignited, dissolved in acid and determined iodometrically.

2.10.2 IS 228 (Part 21) : 2003 ‘Methods for chemical analysis of steels: Part 21 Determination of copper by spectrophotometric method (for copper 0.02 to 0.50 percent)’

Scope — This standard (Part 21) describes the spectrophotometric (diethyldithiocarbamate) method for determination of copper in the range from 0.02 to 0.50 percent in steel.

Outline of the method — The sample is dissolved in acids. A suitable aliquot is taken and copper is complexed with diethyl-dithiocarbamate, extracted in chloroform
and determined spectrophotometrically at 435 nm.

2.11 Tungsten

2.11.1 IS 228 (Part 16) : 1992 ‘Methods for chemical analysis of steels: Part 16 Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent) (second revision)’

Scope — This standard (Part 16) describes spectrophotometric method for determination of tungsten in the range 0.1 to 2 percent in alloy steels.

Outline of the method — In presence of potassium thiocyanate, tungsten in hydrochloric acid is reduced with titanium chloride and stannous chloride to form yellowish-green tungsten thiocyanate complex and measured at 400 nm.

2.12 Nitrogen

2.12.1 IS 228 (Part 17) : 1998 ‘Methods for chemical analysis of steels: Part 17 Determination of nitrogen by thermal conductivity method (for nitrogen up to 0.04 percent) (second revision)’

Scope — This standard (Part 17) covers the determination of nitrogen in the range up to 0.04 percent in steels.

Outline of the method — The nitrogen in steel is converted to molecular nitrogen by inert gas fusion at temperatures above 2 000ºC. Nitrogen in a carrier gas of helium is separated from other gases such as hydrogen and oxygen and is measured by change in thermal conductivity.

2.12.2 IS 228 (Part 19) : 1998 ‘Methods for chemical analysis of steels: Part 19 Determination of nitrogen by steam distillation method (for nitrogen 0.002 to 0.50 percent) (second revision)’

Scope — This standard (Part 19) describes the method for the determination of nitrogen in the range from 0.002 to 0.50 percent in steels.

Outline of the method — The sample is washed thoroughly dried and decomposed with sulphuric acid in the presence of potassium sulphate and a little amount of copper sulphate. It is then steam distilled in the presence of sodium hydroxide and tartaric acid. The nitrogen present is estimated by titrating the distillate with standard sulphuric acid using mixed indicator (bromocresol green and methyl red).

2.12.3 IS 228 (Part 23) : 2003 ‘Methods for chemical analysis of steels: Part 23 Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)’

Scope — This standard (Part 23) covers the determination of nitrogen (0.002 – 1.0 percent) in steel by optical emission spectrometer.

Outline of the method — A capacitor discharge is produced between polished surface of the sample and a conical shaped electrode. The discharge is terminated at a predetermined time. The radiant energy of the nitrogen line and an internal standard line are converted into electrical energy by means of photomultiplier tubes. The concentration of nitrogen in steel is obtained by reference to standard.

2.12.4 IS 228 (Part 24) : 2003 ‘Methods for chemical analysis of steels: Part 24 Determination of total nitrogen in steel by inert gas fusion — thermal conductivity method (nitrogen 0.001 to 0.2 percent)’

Scope — This standard (Part 24) covers the determination of nitrogen in steel in the range 0.001 to 0.2 percent.

Outline of the method — The sample, contained in a small graphite crucible, is fused under a flowing helium atmosphere. Nitrogen present in the steel is released as molecular nitrogen into the flowing helium stream. The nitrogen is separated from other liberated gases such as hydrogen and carbon monoxide and finally measured by thermal conductivity.

2.13 Oxygen

2.13.1 IS 228 (Part 18) : 1998 ‘Methods for chemical analysis of steels: Part 18 Determination of oxygen by instrumental method (for oxygen 0.001 to 0.100 00 percent) (second revision)’

Scope — This standard (Part 18) describes the method for determination of oxygen in steel in the range from 0.001 to 0.100 00 percent by available instruments.

Outline of the method — The sample is melted in a graphite crucible under inert gas stream, at a temperature of not less than 2 000ºC to release oxygen, which combines with carbon from the crucible to form carbon monoxide and which is carried along with the inert gas to infrared detector. The detector output is displayed as oxygen content. The
detector output is calibrated with similar standards for which certified oxygen values are available.

2.14 Hydrogen

2.14.1 IS 228 (Part 22) : 2003 ‘Methods for chemical analysis of steels: Part 22 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)’

Scope — This standard (Part 22) covers the determination of total (diffusible and residual) hydrogen in steel (0.1 ppm to 50 ppm).

Outline of the method — In steel the total hydrogen is present in two forms as diffusible hydrogen and the residual hydrogen. The diffusible hydrogen is the hydrogen which diffuses out during the solidification of the sample. This is contained within the sample tube. During the analysis for the diffusible hydrogen the sample tube is pierced and the diffused hydrogen is swept out with nitrogen and measured. The residual hydrogen is the hydrogen which is remaining after the solidification of sample. This hydrogen is released by heating the sample in a nitrogen atmosphere. Carbon dioxide and moisture released along with hydrogen are removed by suitable absorbent. Hydrogen is then measured by thermal conductivity method.

ANNEX A
(Foreword)

COMMITTEE COMPOSITION

Wrought Steel Products Sectional Committee, MTD 4

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<tr>
<td>Tata Steel Ltd., Jamshedpur</td>
<td>Dr. D. Bhattacharjee (Chairman)</td>
</tr>
<tr>
<td>All India Induction Furnace Association, New Delhi</td>
<td>Sri Indranil Chakraborty (Alternate I)</td>
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<tr>
<td>Bharat Heavy Electricals Ltd., Tiruchirapalli</td>
<td>Dr. A. N. Bhagat (Alternate II)</td>
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<tr>
<td>Central Boilers Board, New Delhi</td>
<td>Sri R. P. Varshney</td>
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<tr>
<td>DGS &amp; D, Bhilai Nagar/Delhi</td>
<td>Sri V. Rajasekharan</td>
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<tr>
<td>Escorts Knowledge Management Centre, Faridabad</td>
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<td>Essar Steels Ltd, Hazira</td>
<td>Sri Alok Nayar</td>
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<td>Jindal South West Ltd., Vasind</td>
<td>Dr. A. K. Das</td>
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<td>M. N. Dastur &amp; Co Ltd, Kolkata/Delhi</td>
<td>Sri R. K. Balasubramaniam (Alternate)</td>
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<td>Ministry of Defence (DGOFB), Kolkata</td>
<td>Sri M.K. Masheshwari</td>
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<td>Ministry of Defence (DGQA), Ichapur</td>
<td>Sri Subhadrata Sengupta</td>
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<td>Ministry of Railways (RDSO), Lucknow</td>
<td>Sri V. K. Tyagi (Alternate)</td>
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<td>Ministry of Steel (Government of India), New Delhi</td>
<td>Sri P. S. Bandhopadhiyay</td>
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<td>Powergrid Corporation of India Ltd., Gurgaon</td>
<td>Sri T. Basu (Alternate)</td>
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<td>Sri S. K. Khilnaney</td>
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<td>SAIL, Bhilai Steel Plant, Bhilai</td>
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Organization

SAIL, Bokaro Steel Plant, Bokaro
SAIL, Central Marketing Organization, Kolkata
SAIL, Durgapur Steel Plant, Durgapur
SAIL, Research & Development Center for Iron & Steel, Ranchi
SAIL, Rourkela Steel Plant, Rourkela
Steel Furnace Association of India, New Delhi
Steel Re-rolling Mills Association of India, Mandi Gobindgarh
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Institute of Steel Development and Growth, Kolkata
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BIS Directorate General

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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 4 (4854).

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Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

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