भारतीय मानक Indian Standard

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 1 आयतनात्मक पद्धति द्वारा कुल कार्बन का निर्धारण (कार्बन 0.05 से 2.50 प्रतिशत के लिए (चौथा पुनरीक्षण)

Methods for Chemical Analysis of Steels Part 1 Determination of Carbon by Volumetric Method (For Carbon 0.05 to 2.50 Percent)

(Fourth Revision)

ICS 77.080.20

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भारतीय मानक ब्यूरो BUREAU OF INDIAN STANDARDS मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002 MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI - 110002 www.bis.gov.in www.standardsbis.in

November 2024

Price Group 6

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 1) (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Chemical analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1952 and subsequently revised in 1959, 1972 and 1987. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This standard covers chemical analysis of both plain carbon and low alloy steels, along with pig iron and cast iron. This part covers the methods for determination of carbon. The other parts of this series are:

Part 2	Determination of manganese in plain carbon and low alloy steels by arsenite method
Part 3	Determination of phosphorus by alkalimetric method
Part 4	Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent)
Part 5	Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent)
Part 6	Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent)
Part 7	Determination of molybdenum by alpha benzoinoxime method (for molybdenum 1 percent and not containing tungsten)
Part 8	Determination of silicon by gravimetric method (for silicon 0.05 to 5.00 percent)
Part 9	Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent)
Part 10	Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)
Part 11	Determination of silicon by reduced molybdosilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent)
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)
Part 13	Determination of arsenic
Part 14	Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)
Part 15	Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent)
Part 16	Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)
Part 17	Determination of nitrogen by thermal conductivity method
Part 18	Determination of oxygen by instrumental method (for oxygen 0.001 to 0.100 0 percent)
Part 19	Determination of nitrogen by steam distillation
Part 20	Determination of carbon and sulphur by infrared absorption method
Part 21	Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent)
	(Continued on third cover)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS PART 1 DETERMINATION OF CARBON BY VOLUMETRIC METHOD (FOR CARBON 0.05 TO 2.50 PERCENT)

(Fourth Revision)

1 SCOPE

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

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	Title
IS 264 : 2005	Nitric acid — Specification
	(third revision)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 6226 (Part 1) : 1994	Recommendations for apparatus for chemicals analysis of metals: Part 1 Apparatus for determination of carbon by direct combustion (<i>first revision</i>)

3 SAMPLING

The sample shall be drawn as prescribed in the relevant Indian Standard. (The sample is cleaned with organic solvent like ether or acetone, dried in an air oven at 100 °C \pm 5 °C before use).

4 APPARATUS

The apparatus recommended in IS 6226 (Part 1) may be used.

5 DETERMINATION OF CARBON BY VOLUMETRIC METHOD

5.1 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.

5.2 Procedure

5.2.1 Before use the apparatus should be tested for satisfactory working against standard steel of appropriate values of carbon.

5.2.2 For Plain Carbon Steel

Take one gram of an accurately weighed and clean sample free from extraneous carbon in the form of small drillings or shavings in a porcelain boat which can withstand a temperature of 1 150 °C without breaking or cracking.

5.2.2.1 Introduce the boat into the hot combustion tube in the furnace kept at 1 000 °C to 1 100 °C.

5.2.3 For Low Alloy and High Alloy Steels

Take one gram of an accurately weighed and clean sample free from extraneous carbon in the form of small drillings or shavings in a porcelain boat, which can stand a temperature of 1 250 °C without breaking and cracking. Spread 0.5 g of pure tin granules over the sample. In case of high alloy steel mix the sample with 0.5 g of pure iron (99.99 percent) filings also. Introduce the boat into the hot combustion tube in the furnace, kept between 1 150 °C to 1 250 °C.

5.2.4 Close the furnace inlet with a rubber stopper, allow the sample to heat for one to one and a half minute. Regulate the flow of oxygen to 300 ml to 400 ml per minute into the furnace and establish connection with the burette, which has been previously filled with acidulated water/brine water coloured with methyl red, so that the liquid level in the bulbed portion of the gas burette does not fall rapidly. After a minute or so the level of water in the burette begins to fall more rapidly, though the same rate of oxygen is maintained, indicating completion of combustion.

5.2.5 Take readings, when the level reaches near the zero graduation mark after closing the bend way stopcock and equalizing the levels of the burette and the connected levelling bottle. Pass the collected and measured gas twice into the absorbing bulb, till constant reading is obtained, Record the burette reading. On the basis of one gram of sample taken for analysis, the burette is graduated to measure directly the percentage of carbon.

5.2.5.1 Examine the combustion boat for complete fusion of the sample, if not thoroughly fused, repeat the determination with a fresh sample.

5.3 Blank

Run a blank experiment on the same quantity of accelerators used, without any sample and make the appropriate corrections.

5.4 Calculation

Carbon, percent = $(A - B) \times F$

where

- A = burette reading after absorption of carbon dioxide in caustic potash with one gram of sample;
- B = burette reading for the blank experiment; and
- F = correction teeter for temperature and pressure (*see* Table 1).

5.5 Reproducibility

- a) ± 0.01 percent up to 1.50 percent carbon; and
- b) ± 0.02 percent above 1.50 percent carbon.

Table 1 Correction Factors

|--|

SI No.	Pressure,	700	702	704	706	708	710	712	714	716	718	720	722	724	726	728	730	732	734
	mm Hg Temperature , ℃																		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)
i)	15	0.924	0.927	0.929	0.932	0.935	0.937	0.940	0.943	0.945	0.948	0.951	0.954	0.956	0.959	0.962	0.964	0.967	0.970
ii)	16	0.920	0.922	0.925	0.928	0.930	0.930	0.936	0.938	0.941	0.944	0.946	0.949	0.952	0.954	0.957	0.960	0.962	0.965
iii)	17	0.915	0.918	0.921	0.923	0.926	0.929	0.931	0.934	0.937	0.939	0.942	0.945	0.947	0.950	0.953	0.955	0.958	0.961
iv)	18	0.911	0.914	0.916	0.919	0.922	0.924	0.927	0.929	0.932	0.935	0.937	0.940	0.943	0.945	0.948	0.951	0.953	0.956
v)	19	0.906	0.909	0.912	0.914	0.917	0.920	0.922	0.925	0.928	0.930	0.933	0.936	0.938	0.941	0.944	0.946	0.949	0.952
vi)	20	0.902	0.904	0.907	0.910	0.912	0.915	0.918	0.920	0.923	0.926	0.928	0.931	0.934	0.936	0.939	0.942	0.944	0.947
vii)	21	0.897	0.900	0.903	0.905	0.908	0.910	0.913	0.916	0.918	0.921	0.924	0.926	0.929	0.932	0.934	0.937	0.940	0.942
viii)	22	0.893	0.895	0.898	0.901	0.903	0.906	0.908	0.911	0.914	0.916	0.919	0.922	0.924	0.927	0.930	0.932	0.935	0.937
ix)	23	0.888	0.891	0.893	0.896	0.899	0.901	0.904	0.906	0.909	0.912	0.914	0.917	0.912	0.922	0.925	0.927	0.930	0.933
x)	24	0.883	0.886	0.889	0.891	0.894	0.896	0.899	0.902	0.904	0.907	0.910	0.912	0.915	0.917	0.920	0.922	0.925	0.928
xi)	25	0.879	0.881	0.884	0.886	0.889	0.892	0.894	0.897	0.900	0.902	0.905	0.907	0.910	0.912	0.915	0.918	0.920	0.923
xii)	26	0.874	0.876	0.879	0.882	0.884	0.887	0.889	0.892	0.894	0.897	0.900	0.902	0.905	0.908	0.910	0.913	0.915	0.918
xiii)	27	0.869	0.872	0.874	0.877	0.879	0.882	0.884	0.887	0.890	0.892	0.895	0.897	0.900	0.902	0.905	0.908	0.910	0.913
xiv)	28	0.864	0.867	0.869	0.872	0.874	0.877	0.879	0.882	0.885	0.887	0.890	0.892	0.895	0.898	0.900	0.903	0.905	0.908
xv)	29	0.859	0.862	0.864	0.867	0.869	0.872	0.874	0.877	0.880	0.882	0.885	0.887	0.890	0.892	0.895	0.897	0.900	0.903
xvi)	30	0.854	0.856	0.859	0.862	0.864	0.867	0.869	0.872	0.874	0.877	0.879	0.882	0.884	0.887	0.890	0.892	0.895	0.897
xvii)	31	0.849	0.851	0.854	0.856	0.859	0.861	0.864	0.866	0.869	0.872	0.874	0.877	0.879	0.882	0.884	0.887	0.889	0.892
xviii)	32	0.843	0.846	0.848	0.851	0.854	0.856	0.859	0.861	0.864	0.866	0.869	0.871	0.874	0.876	0.879	0.882	0.884	0.886
xix)	33	0.838	0.840	0.843	0.846	0.848	0.851	0.853	0.856	0.858	0.861	0.863	0.866	0.868	0.871	0.873	0.876	0.878	0.881
xx)	34	0.833	0.835	0.838	0.840	0.843	0.845	0.848	0.850	0.853	0.855	0.858	0.860	0.863	0.865	0.868	0.870	0.873	0.875
xxi)	35	0.827	0.830	0.832	0.834	0.837	0.840	0.842	0.845	0.847	0.850	0.852	0.855	0.857	0.860	0.862	0.865	0.867	0.870
xxii)	36	0.821	0.824	0.826	0.829	0.831	0.834	0.836	0.839	0.841	0.844	0.846	0.849	0.851	0.854	0.856	0.859	0.861	0.864
xxiii)	37	0.816	0.818	0.820	0.823	0.826	0.828	0.830	0.833	0.836	0.838	0.840	0.843	0.846	0.848	0.850	0.853	0.856	0.858

							Т	able 1 (C	Conclude	<i>d</i>)									
Sl No.	Pressure, mm Hg Temperature, ℃	700	702	704	706	708	710	712	714	716	718	720	722	724	726	728	730	732	734
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)
xxiv)	38	0.810	0.812	0.815	0.817	0.820	0.822	0.825	0.827	0.830	0.832	0.834	0.837	0.840	0.842	0.844	0.847	0.850	0.852
xxv)	39	0.804	0.806	0.809	0.811	0.814	0.816	0.818	0.821	0.824	0.826	0.828	0.831	0.833	0.836	0.838	0.841	0.843	0.846
xxvi)	40	0.798	0.800	0.802	0.805	0.807	0.810	0.812	0.815	0.817	0.820	0.822	0.825	0.827	0.830	0.832	0.835	0.837	0.840
xxvii)	41	0.791	0.794	0.796	0.799	0.801	0.804	0.806	0.808	0.811	0.813	0.816	0.818	0.821	0.823	0.826	0.828	0.831	0.833
xxviii)	42	0.785	0.787	0.790	0.792	0.795	0.797	0.800	0.802	0.804	0.807	0.809	0.812	0.814	0.817	0.819	0.822	0.824	0.827
xxix)	43	0.778	0.781	0.783	0.786	0.788	0.791	0.793	0.796	0.798	0.800	0.803	0.805	0.808	0.810	0.813	0.815	0.818	0.820
xxx)	44	0.772	0.774	0.776	0.779	0.781	0.784	0.786	0.789	0.791	0.794	0.796	0.798	0.801	0.803	0.806	0.808	0.811	0.813
xxxi)	45	0.765	0.761	0.770	0.772	0.775	0.777	0.779	0.782	0.784	0.787	0.789	0.792	0.794	0.796	0.799	0.801	0.804	0.806
								\bigcirc											
Sl No.	Pressure,	736	738	740	742	744	746	748	750	752	754	756	758	760	762	764	766	768	770
	mm Hg Temperature , °C																		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)
i)	15	0.972	0.975	0.978	0.980	0.983	0.986	0.988	0.991	0.994	0.996	0.999	1.002	1.005	1.007	1.010	1.013	1.015	1.018
ii)	16	0.968	0.970	0.973	0.976	0.978	0.981	0.984	0.987	0.989	0.992	0.995	0.997	1.000	1.003	1.005	1.008	1.011	1.013
iii)	17	0.963	0.966	0.969	0.971	0.974	0.977	0.979	0.982	0.985	0.987	0.990	0.993	0.995	0.998	1.001	1.003	1.006	1.009
iv)	18	0.959	0.961	0.964	0.967	0.969	0.972	0.975	0.977	0.980	0.983	0.985	0.988	0.991	0.993	0.996	0.999	1.001	1.004
v)	19	0.954	0.957	0.959	0.962	0.965	0.967	0.970	0.973	0.975	0.978	0.981	0.983	0.986	0.989	0.991	0.994	0.996	0.999
vi)	20	0.950	0.952	0.955	0.957	0.960	0.963	0.965	0.968	0.971	0.973	0.976	0.978	0.981	0.984	0.986	0.989	0.992	0.994
vii)	21	0.945	0.947	0.950	0.953	0.955	0.958	0.961	0.963	0.966	0.968	0.971	0.974	0.976	0.979	0.982	0.984	0.987	0.990
viii)	22	0.940	0.943	0.945	0.948	0.950	0.953	0.956	0.958	0.961	0.964	0.966	0.969	0.972	0.974	0.977	0 979	0.982	0.985
ix)	23	0.935	0.938	0.940	0.943	0.946	0.948	0.951	0.954	0.956	0.959	0.961	0.964	0.967	0.969	0.972	0.974	0.977	0.980
x)	24	0.930	0.933	0.936	0.938	0.941	0.943	0.946	0.949	0.951	0.954	0.956	0.959	0.962	0.964	0.967	0.967	0.972	0.975
xi)	25	0.925	0.928	0.931	0.933	0.936	0.938	0.941	0.944	0.946	0.949	0.951	0.954	0.957	0.959	0.962	0.964	0.967	0.970

							\mathcal{D}												
Sl No.	Pressure, mm Hg Temperature,	736	738	740	742	744	746	748	750	752	754	756	758	760	762	764	766	768	770
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)
xii)	26	0.920	0.923	0.926	0.928	0.931	0.933	0.936	0.939	0.941	0.944	0.946	0.949	0.952	0.954	0.957	0.959	0.962	0.964
xiii)	27	0.915	0.918	0.921	0.923	0.926	0.928	0.931	0.934	0.936	0.939	0.941	0.944	0.946	0.949	0.952	0.954	0.957	0.959
xiv)	28	0.910	0.913	0.916	0.918	0.921	0.923	0.926	0.928	0.931	0.934	0.936	0.939	0.941	0.944	0.946	0.949	0.952	0.954
xv)	29	0.905	0.908	0.910	0.913	0.915	0.918	0.920	0.923	0.926	0.928	0.931	0.933	0.936	0.938	0.941	0.944	0.946	0.949
xvi)	30	0.900	0.902	0.905	0.908	0.910	0.913	0.915	0.918	0.920	0.923	0.925	0.928	0.930	0.933	0.936	0.938	0.941	0.943
xvii)	31	0.894	0.897	0.900	0.902	0.905	0.907	0.910	0.912	0.915	0.917	0.920	0.922	0.925	0.928	0.930	0.933	0.935	0.938
xviii)	32	0.889	0.892	0.894	0.897	0.899	0.902	0.904	0.907	0.909	0.912	0.914	0.917	0.920	0.922	0.925	0.927	0.930	0.932
xix)	33	0.884	0.886	0.889	0.891	0.894	0.896	0.899	0.901	0.904	0.906	0.909	0.911	0.914	0.916	0.919	0.922	0.924	0.927
xx)	34	0.878	0.880	0.883	0.886	0.888	0.891	0.893	0.896	0.898	0.901	0.903	0.906	0.908	0.911	0.913	0.916	0.918	0.921
xxi)	35	0.872	0.875	0.877	0.880	0.882	0.885	0.887	0.890	0.892	0.895	0.897	0.900	0.902	0.905	0.907	0.910	0.912	0.915
xxii)	36	0.866	0.869	0.871	0.874	0.876	0.879	0.882	0.884	0.886	0.889	0.892	0.894	0.896	0.899	0.902	0.904	0.906	0.909
xxiii)	37	0.860	0.863	0.866	0.868	0.870	0.873	0.876	0.878	0.880	0.883	0.886	0.888	0.890	0.893	0.896	0.898	0.900	0.903
xxiv)	38	0.854	0.857	0.859	0.862	0.864	0.867	0.869	0.872	0.874	0.877	0.879	0.882	0.884	0.887	0.889	0.892	0.894	0.897
xxv)	39	0.848	0.851	0.853	0.856	0.858	0.861	0.863	0.866	0.868	0.871	0.873	0.876	0.878	0.881	0.883	0.886	0.888	0.890
xxvi)	40	0.842	0.844	0.847	0.850	0.852	0.854	0.857	0.859	0.862	0.864	0.867	0.869	0.872	0.874	0.877	0.879	0.882	0.884
xxvii)	41	0.836	0.838	0.841	0.843	0.846	0.848	0.850	0.853	0.855	0.858	0.860	0.863	0.865	0.868	0.870	0.873	0.875	0.878
xxviii)	42	0.829	0.832	0.834	0.836	0.839	0.841	0.844	0.846	0.849	0.851	0.854	0.856	0.859	0.861	0.864	0.866	0.868	0.870
xxix)	43	0.822	0.825	0.827	0.830	0.832	0.835	0.837	0.840	0.842	0.844	0.847	0.849	0.852	0.854	0.857	0.859	0.862	0.864
xxx)	44	0.816	0.818	0.820	0.823	0.825	0 828	0.830	0.833	0.835	0.838	0.840	0.842	0.845	0.847	0.850	0.852	0.855	0.857
xxxi)	45	0.809	0.811	0.814	0.816	0.818	0.821	0.823	0.826	0.828	0.830	0.833	0.835	0.838	0.840	0.843	0.845	0.848	0.850

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

Organization

CSIR - National Metallurgical Laboratory, Jamshedpur Arcelor Mittal Nippon Steel, Mumbai

Bhabha Atomic Research Centre, Mumbai

CSIR - National Metallurgical Laboratory, Jamshedpur

Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad

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Geological Survey of India, New Delhi

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Indian Metals and Ferro Alloys Limited, Bhubaneswar

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Member Secretary Shri Ashish Prabhakar Wakle Scientist 'D'/Joint Director (Metallurgical Engineering), BIS this Page has been intertionally left blank

- Part 22 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)
- Part 23 Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)
- Part 24 Determination of nitrogen in steel by inert gas fusion Thermal conductivity method (nitrogen 0.001 to 0.2 percent)

The composition of the Committee responsible for the formulation of this standard is given in <u>Annex A</u>.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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