Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2 [ Ref : Doc : SMDC 2 ( 3265

Indian Standard

## METHODS FOR CHEMICAL ANALYSIS OF STEELS

# PART 1 DETERMINATION OF CARBON BY VOLUMETRIC METHOD (FOR CARBON 0.05 TO 2.50 PERCENT)

(Third Revision)

1. 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.

#### 2. Determination of Carbon

- 2.1 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 burrette, the contraction in volume is read against a scale, calibrated directly to the percentage of carbon.
- 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  $100^{\circ} \pm 5^{\circ}$ C before use.)
- 4. Apparatus The apparatus recommended in IS: 6226 (Part 1)-1971 'Recommendations for apparatus for chemical analysis of metals: Part 1 Determination of carbon by direct combustion method' may be used.

#### 5. Procedure

- 5.1 Before use the apparatus should be tested for satisfactory working against standard steel of appropriate values of carbon.
- 5.2 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.
- **5.2.1** Introduce the boat into the hot combustion tube in the furnace kept at  $1\,000^\circ$  to  $1\,100^\circ$ C.
- 5.3 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° to 1 250°C.
- 5.4 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 300-400 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.
- 5.5 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

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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.

- **5.5.1** Examine the combustion boat for complete fusion of the sample, if not thoroughly fused, repeat the determination with a fresh sample.
- 5.6 Blank Run a blank experiment on the same quantity of accelerators used, without any sample and make the appropriate corrections.
- 5.7 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 factor for temperature and pressure ( see Table 1 ).
- 5.8 Reproducibility  $\pm 0.01$  percent up to 1.50 percent carbon, and  $\pm 0.02$  percent above 1.50 percent carbon.

### APPENDIX A

## INDIAN STANDARDS ON METHODS FOR CHEMICAL ANALYSIS OF STEELS

- IS: 228 Methods for chemical analysis of steels:
  - (Part 2)-1987 Determination of manganese in plain carbon and low alloy steels by arsenite method (third revision)
  - (Part 3)-1987 Determination of phosphorus by alkalimetric method (third revision)
  - (Part 4)-1987 Determination of carbon by gravimetric method (for carbon ≥ 0·1 percent) (third revision)
  - ( Part 5 )-1987 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ≥ 0.1 percent) (third revision)
  - (Part 6)-1987 Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent) (third revision)
  - (Part 7)-1974 Determination of molybdenum by α-benzoinoxime method (for molybdenum > 1 percent) (second revision)
  - (Part 8)-1975 Determination of silicon by the gravimetric method (for silicon ≥ 0.1 percent) (second revision)
  - (Part 9)-1975 Determination of sulphur in plain carbon steels by evolution method (second revision)
  - (Part 10)-1976 Determination of molybdenum by thiocyanate (photometric) method (for molybdenum up to 1 percent) in low and high alloy steels (second revision)
  - ( Part 11 )-1976 Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent) ( second revision)
  - (Part 12)-1988 Determination of manganese by periodate (spectrophotometric) method in low and high alloy steels (for manganese up to 0.01 to 2.00 percent) (second revision)
  - (Part 13)-1982 Determination of arsenic
  - (Part 14 )-1987 Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)

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Pressure,																					
mm Hg												•									
	730	732	734	736	738	740	742	744	746	748	750	752	754	756	758	760	762	764	766	768	770
									, 40	740	,,,,	752	754	750	750	700	702	704	700	700	770
Tempera-																					
ture, °C																					
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)	(21)	(22)
15	0.964	0.967	0.970	0.972	2 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
16	0.960	0.962	0.965	0.968	3 0.970	ე∙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		
17				0.963	3 0 <sup>,</sup> 966	0.969	0.971	0.974	0.977	0.979	0.982	0.985	0.987					1.001		1.006	
18				0.959			0.967	0.969	0.972	0.975	0.977	0.980	0.983	0.985	0.988	0.991	0.993		0.999		1.004
19	0.946	0.949	0.952	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
20	0.942	0.944	0.947	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
21	0.937	0.940	0.942	0.945	0.947	0.950	0.953	0.955	0.958	0.961	0.963	0.966		0.971					0.984	0.987	0.990
22	0.932	0.935	0.937	0.940	0.943	0.945	0.948	0.950	0.953	0.956	0.958	0.961	0.964	0.966					0.979	0.982	0.985
23		0.930	0.933	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
24	0.922	0.925	0.928	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
25	0.918	0.920	0.923	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
26	0.913	0.915	0.918	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	
27					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
28	0.903	0.902	0.908	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
29	0.897	0.900	0.903	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
30	0.892	0.895	0.897	0.900	0.902	0.905	0.908	0.910	0.913	0.915	0.918	0.920	0 923	0.925	0.028	0.930	0.033	0.936	0.938	0.941	0.943
31					0.897		0.902	0.905	0.907	0.910	0.912	0.915	0.923	0 920	0.920				0.933	0.935	0.938
32					0.892		0.897	0.899	0.902	0.904	0.907	0.909	0.912	0.914						0.930	0.932
33	0.876	0.878	0.881	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
34	0.870	<b>0</b> ·873	0.875	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	ŏ∙911	0.913	0.916	0.918	0.921
35	0.865	0.867	0.870	0.872	0.875	0.877	0.880	0.882	0.885	0.887	იაგიი	0.892	0.895	0.807	0.900	0.902	0.905	0.907	0.910	0.912	0.915
36	0.859	0.861	0.864	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		0.904		0.909
37	0.853	0.856	0.858	0.860	0.863	0.866	0.868	0.870	0.873	0.876	0.878	0.880	0.883	0.886				0.896		0.900	0.903
38	0.847	0.850	0.852	0.854	0.857	9 859	0.862	0.864	0.867	0.869	0.872	0.874	0.877	0.879	0.885	0.884	0.887				0.897
39	0.841	0.843	0.846	0 <sup>.</sup> 848	0.851	0.823	0.856	0.858	0.861	0.863	0.866	0.868	0.871	0.873	0.876	0.878	0.881			0.888	0.890
40	0.835	0.837	0.840	0.842	0.844	0.847	0.850	0:852	0.854	0.857	0.850	0.863	0.864	0.867	0.880	0.872	0.874	0.877	0.879	0.882	0.884
41	n.828	0.831	0.833	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				0.878
42	Ŏ·822	0.824	0.827	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					
43	0.815	0.818	0.820	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
44	808.0	0.811	0.813	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				0.857
45	0.801	0.804	0.806	0.800	0.014	0.01 4											0.040	0.040	0.045	0.040	0.050
40	001	0 004	0 000	0 009	0.811	U'814	0.016	0.918	0.821	0.823	0.876	0.858	0.830	0.833	0.832	0.838	0.840	0.843	0.845	บ•848	0.850

## **EXPLANATORY NOTE**

IS: 228 was issued as a tentative standard in 1952 and revised in 1959 covering the chemical analysis of pig iron, cast iron and plain carbon and low alloy steels. For the convenience, it was decided to publish comprehensive series on chemical analysis of steels including high alloy steels and another series on chemical analysis of pig iron and cast iron. Accordingly, chemical analysis of steels was published in various parts. This standard is series of parts on chemical analysis of steels. The other parts published are given in Appendix A. The chemical analysis of pig iron and cast iron is being published in separate standard.

In this revision the major modifications are as follows:

- a) The limit of determination of carbon in steel have been modified as 0.05 to 2.50 percent in place of greater than or equal to 0.1 percent.
- b) The range of pressure for the correction factors in Table 1 have been incorporated from 730 to 770 instead of 700 to 770.