*भारतीय मानक*

# इस्पात के रासायनिक विश्लेषण की प्रणाली

## भाग 18 मापयन्त्री प्रणाली द्वारा आक्सीजन का निर्धारण ( 0.001 से 0.100 0 प्रतिशत आक्सीजन के लिए )

*( दूसरा पुनरीक्षण )*

*Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF STEELS

## PART 18 DETERMINATION OF OXYGEN BY INSTRUMENTAL METHOD ( FOR OXYGEN 0.001 to 0.100 0 PERCENT )

*( Second Revision )*

ICS 77.080.20

BUREAU OF INDIAN STANDARDS
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Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

## FOREWORD

This Indian Standard (Part 18) (Second Revision) 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 228, which was first published in 1952 and subsequently revised in 1959, covered the chemical analysis of plain carbon and low alloy steels, along with pig iron and cast iron. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts.

This part covers the methods for determination of oxygen. The other parts of this series are:

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|---|---|
| IS 228 | Methods for chemical analysis of steels : |
| (Part 1) : 1987 | Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent) |
| (Part 2) : 1987 | Determination of manganese in plain carbon and low alloy steels by arsenite method |
| (Part 3) : 1987 | Determination of phosphorus by alkalimetric method |
| (Part 4) : 1987 | Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent) |
| (Part 5) : 1987 | Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent) |
| (Part 6) : 1987 | Determination of chromium by persulphate oxidation method (for chromium greater than or equal to 0.1 percent) |
| (Part 7) : 1990 | Determination of molybdenum by benzoinoxine method (for molybdenun greater than or equal to 1 percent) |
| (Part 8) : 1989 | Determination of silicon by the gravimetric method (for silicon 0.05 to 5.00 percent) |
| (Part 9) : 1989 | Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent) |
| (Part 10) : 1989 | Determination of molybdenum by thiocyanate (photometric ) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent) |
| (Part 11) : 1990 | Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent) |
| (Part 12) : 1988 | Determination of manganese by periodate spectrophotometric method in low and high alloy steels (for manganese 0.01 to 2.0 percent) |
| (Part 13) : 1982 | Determination of arsenic |
| (Part 14) : 1988 | Determination of carbon by themal conductivity method (for copper 0.005 to 2.000 percent) |
| (Part 15) : 1992 | Determination of copper by thiosulaphate iodide method (for copper 0.05 to 5 percent) |
| (Part 16) : 1992 | Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent) |
| (Part 17) : 1998 | Determination of nitrogen by thermal conductivity method (for nitrogen up to .04 percent) |

In this revision 'Instrumental Method' for determination of oxygen in steel has been introduced.

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 same as that of the specified value in this standard.

*Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF STEELS

## PART 18 DETERMINATION OF OXYGEN BY INSTRUMENTAL METHOD ( FOR OXYGEN 0.001 to 0.100 0 PERCENT )

*( Second Revision )*

### 1 SCOPE

This standard (Part 18) describes the method for determination of oxygen in steel in the range from 0.001 to 0.100 0 percent by available instruments.

### 2 SAMPLING

**2.1** Location and selection of sample can be as per requirements and to get a good representative sample. Use only solid samples to minimise the errors due to surface oxidation. Sample size and weight shall be such so as to suit the crucible in which fusion is being carried out.

**2.2** Sample preparation technique is critical to attain consistent, reliable and reproducible oxygen readings. Cut samples to the appropriate size using cut-off wheels or by machining. Avoid over heating and other oxide contaminations during preparation. Typical sample size of 5-6 mm dia (or 5 $mm^2$) and 50 mm length is suitable for all types of instruments. File the piece with clean, smooth file and abrade the entire surface to remove all traces of oxidation. Cut by using clean hacksaw blade (without paint) to 3-4 mm long pieces weighing approx 1 g. Hand file again the cut faces. With a fine grade silicon carbide paper clean the surface and drop it in a bottle containing acetone and do ultrasonic cleaning. While handing the sample use tweezers. Do not touch the surface with fingers during and in the following stages of cleaning.

### 3 DETERMINATION OF OXYGEN

#### 3.1 Outline of the Method

The sample is melted in a graphite crucible under inert gas stream, at a temperature of not less than 2 000°C 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.

#### 3.2 Instruments

**3.2.1** For the instruments based on the infrared detector the sample is melted in the furnace in a stream of argon helium or nitrogen depending upon the instrument model and the evolved gases are passed through infrared detector where CO is measured directly or after its conversion to $CO_2$. The detector response to this change is displayed directly as oxygen content.

#### 3.3 Reagents

**3.3.1** *Acetone or* n-*Hexane*

**3.3.2** *Ascarite*

**3.3.3** *Inert Gas Helium, Argon of the Required Purity*

**3.3.4** *Magnesium Perchlorate*

**3.3.5** *Charcoal and Copper Oxide,* in a rare-earth oxide mixed based (used in some instruments).

#### 3.4 Procedure

**3.4.1** Prepare and stabilise the instrument. Change the chemicals and filters as required. Check for leakage and run two or three dummy analysis to check the operation and clean the system before taking up calibration with standard samples.

**3.4.2** Since accuracy of analysis mainly depends on the standards, for calibration select highly homogeneous samples of identical base material, for which oxygen values have been certified. In some instruments, gas dosing calibration is available to set up the instrument response, but it is recommended to confirm the same with standards.

**3.4.3** Determine the blank value as per the instructions and incorporate the same if the instrument is automatic-type or it can be reduced from the oxygen value later.

**3.4.4** Weigh accurately to the nearest 1 mg of calibration standard having low, medium and high oxygen contents. Weight shall be as per the instrument's capability.

**3.4.5** Follow the calibration procedures as laid down by the operation manual of the instrument and establish the instrument response. Confirm the same by running two additional standards. The response should be within ± 0.000 5 percent oxygen for duplicate runs.

**3.4.6** Take freshly prepared sample as per the Sample preparation procedure (or sample prepared and stored in acetone). Wash the same in acetone or n-hexane and dry thoroughly. Weigh the sample accurately and place it in the automatic sample loading device. Carry out analysis as per the procedure specified by the instrument manufacturer. Note the oxygen value. Carry out the analysis in replicate and average the values, discarding the outlier beyond ± 0.000 5 percent. At least three values should be within ± 0.000 5 percent.

### 3.5 Precision

Expected + 0.000 5 at 0.005 0 percent oxygen level.

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This Indian Standard has been developed from Doc: No. MTD 2 ( 4105 ).

### Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
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