

Sampling and analysis of iron, steel and other ferrous metals —

Part 3: Methods of analysis —

Section 3.22 Determination of nitrogen —

Subsection 3.22.1 Steel: volumetric method

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Committees responsible for this British Standard

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- BCIRA
- British Steel Industry
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Ferro Alloys and Metals Producers' Association
- Ministry of Defence

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Foreword

This Subsection of BS 6200 has been prepared under the direction of the Iron and Steel Standards Policy Committee and supersedes method 1 for the determination of nitrogen in BSI Handbook No. 19, to which it is technically equivalent.

BS 6200 is a multipart British Standard, covering all aspects of the sampling and analysis of iron, steel and other ferrous metals. A list of contents, together with general information, is given in Part 1.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Subsection of BS 6200 describes a volumetric method for the determination of nitrogen in steel.

The method is applicable to nitrogen contents from 0.005 % (*m/m*) to 0.25 % (*m/m*) in all types of steel except those containing silicon nitride.

NOTE 1 Complete recovery of nitrogen cannot be obtained from steels containing silicon nitride. This nitride has been found only in samples of silicon steels manufactured without aluminium additions, and then only in sheet material.

NOTE 2 The titles of the publications referred to in this Subsection of BS 6200 are listed on the inside back cover.

2 Principle

After solution of the test portion in hydrochloric acid the “acid soluble” and “insoluble” nitrogen are separated by means of a centrifuge and the insoluble residue is decomposed by fuming strongly with sulfuric acid. The “acid soluble” and “insoluble” nitrogen contents are recovered separately as ammonia by steam distillation over sodium hydroxide and the determinations are completed volumetrically by titration with hydrochloric acid. The total nitrogen content is obtained from the sum of the two determinations.

NOTE The terms “soluble” and “insoluble” nitrogen refer to chemical reactivity under the solvent conditions specified in this method, and do not necessarily imply metallurgical states.

3 Reagents

During the analysis use only reagents of recognized analytical grade which are known to give a very low nitrogen blank. Use only grade 3 water as specified in BS 3978 except where ammonia-free water is specifically stated.

NOTE If a solution requires filtration, wash the filter paper thoroughly with ammonia-free water before use.

3.1 Ammonia-free water. Prepare ammonia-free water by passing distilled water through the cation exchange column (4.4). It is essential to wash the resin column with acid before use to convert it to the hydrogen form. This is conveniently performed by passing 2 l of hydrochloric acid (3.9) through the column, then washing with water until freed from acid.

3.2 Barium chloride solution, 100 g/l. Dissolve 10 g of barium chloride, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in ammonia-free water (3.1), dilute to 100 ml with ammonia-free water and mix.

3.3 Boric acid solution, 1 g/l. Dissolve 1 g of boric acid in water, dilute to 1 l and mix.

3.4 Chromic-sulfuric acid. Dissolve 2 g of chromium(VI) oxide in 50 ml of water and add slowly and with constant stirring, 100 ml of sulfuric acid (3.16).

3.5 Diphenylamine reagent. To 25 ml of water, add cautiously with stirring, 75 ml of sulfuric acid (3.16). Then add 0.1 g of diphenylamine, stir until dissolved and cool.

3.6 Disodium tetraborate, standard solution, 1 ml equivalent to 0.14 mg nitrogen. Dissolve 1.9072 g of freshly recrystallized disodium tetraborate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, in 200 ml of water. Cool, transfer to a 1 l volumetric flask, dilute to the mark and mix.

To prepare recrystallized disodium tetraborate, dissolve 15 g of the reagent in 50 ml of hot water. Cool. Filter the crystals under suction. Wash first with two 10 ml portions of cold water, then with two 10 ml portions of 95 % ethanol, and finally with two 10 ml portions of diethyl ether. Spread the crystals on a clock glass and as soon as the ether has evaporated, transfer them to a weighing bottle and stopper tightly.

3.7 Hydrochloric acid, density $\rho = 1.16$ g/ml to 1.18 g/ml.

3.8 Hydrochloric acid, redistilled. To 300 ml of ammonia-free water (3.1) add 300 ml of hydrochloric acid (3.7) and distil in an all-glass distillation apparatus (4.6). Reject the first 100 ml of distillate and collect the next 400 ml for use.

3.9 Hydrochloric acid, $\rho = 1.16$ g/ml to 1.18 g/ml, diluted 1 + 9.

3.10 Hydrochloric acid, 0.01 M, approximately. Transfer 9.0 ml of hydrochloric acid (3.7) by means of a burette to a 1 l volumetric flask, dilute to the mark and mix. Transfer 100 ml of this solution to a second 1 l volumetric flask, dilute to the mark and mix.

3.11 Hydrofluoric acid, 40 % (*m/m*) approximately.

3.12 Hydrogen peroxide, approximately 6 g per 100 ml (20 volume).

3.13 Screened methyl red indicator. Dissolve 0.125 g of methyl red and 0.083 g of methylene blue in 100 ml of ethanol and mix.

3.14 Sodium hydroxide solution, 400 g/l. Dissolve 400 g of sodium hydroxide in ammonia-free water (3.1), dilute to 1 l with ammonia-free water (3.1) and mix. Prepare this solution in a polyethylene beaker with stirring and cooling. Store in a stoppered polyethylene bottle.

3.15 Sodium sulfate, anhydrous.

3.16 Sulfuric acid, $\rho = 1.84$ g/ml.

Test individual bottles of sulfuric acid and select one with a nitrogen content in any form of less than 0.5 $\mu\text{g/g}$. Nitrogen as ammonia will usually be the major source of contamination, and this may be detected by normal methods. Nitrate may also be present and may be detected by the following procedure.