SLOVENSKI STANDARD
SIST EN 10276-1:2001

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Kemična analiza železnih materialov - Določevanje kisika v jeklu in železovih litinah - 1. del: Vzorčenje in priprava vzorcev za določitev kisika

Chemical analysis of ferrous materials - Determination of oxygen in steel and iron - Part 1: Sampling and preparation of steel samples for oxygen determination

Chemische Analyse von Eisenmetallen - Bestimmung des Sauerstoffgehalts von Stahl und Eisen - Teil 1: Herstellung und Vorbereitung der Stahlproben für die Sauerstoff-Bestimmung

Analyse chimique des matériaux sidérurgiques - Dosage de l'oxygene dans les aciers et les fontes - Partie 1: Echantillonnage et préparation des échantillons en acier pour dosage de l'oxygene

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Chemical analysis of ferrous materials - Determination of oxygen in steel and iron - Part 1: Sampling and preparation of steel samples for oxygen determination

Analyse chimique des matériaux sidérurgiques - Dosage de l'oxygène dans les aciers et les fontes - Partie 1: Echantillonnage et préparation des échantillons en acier pour dosage de l'oxygène

Chemische Analyse von Eisenmetallen - Bestimmung des Sauerstoffgehalts von Stahl und Eisen - Teil 1: Herstellung und Vorbereitung der Stahlproben für die Sauerstoff-Bestimmung

This European Standard was approved by CEN on 22 April 2000.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Contents

Foreword

This European Standard has been prepared by Technical Committee ECISS/TC 20 "Methods of chemical analysis of ferrous products", the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2000, and conflicting national standards shall be withdrawn at the latest by November 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies two methods for the preparation of steel samples for oxygen determination. Both methods are suitable for preparing samples for the determination of oxygen content, especially for oxygen contents < 0,0050 %. This standard is applicable to steels having a hardness of < 400 HBW 10/3000.

2 Normative Reference

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 14284	Steel and iron - Sampling and preparation of samples for the determination of chemical composition

3 Principle

Samples for oxygen determination are machined to a suitable shape and size within the restrictions imposed by the instrument used. In order to ensure that the surface has the minimum possible oxygen content, samples for analysis are prepared either by punching (method A) or by turning (method B).

4 Reagents

4.1 Organic oxygen free hydro carbon solvent e.g. n-hexane; (boiling-point range: 68 °C to 69 °C)

4.2 Argon or nitrogen

5 Apparatus

5.1 Methods A and B

5.1.1 Mechanical steel saw

5.1.2 Tweezers

5.1.3 Receptacles for storing the test samples e.g. glass tubes about 2,5 cm long and about 1 cm in diameter with stoppers/caps.

5.2 Method A:

5.2.1 Punch: suitable for punching out samples in the manner described in 7.1.4.

Purging by argon or nitrogen ensures minimum possible contact of the samples with atmospheric oxygen. This is illustrated in figure 1.

5.2.2 Flat bed linisher for linishing the steel sample.

5.2.3 Electrical hand drill and rotary file, with rotation speed exceeding 30 000 rev/min.

5.3 Method B:

5.3.1 Lathe for turning steel samples at speeds of 250 rev/min to 1 200 rev/min.

5.3.2 Hand saw: the saw blade shall be free from grease or paint.

5.3.3 Filing block made of stainless steel which holds the samples firm during filing of the end-faces (figure 2).

5.3.4 Hand files: cut no. 4.

5.3.5 Metal wire brush with strong bristles about 2 cm long.

6 Sampling

Sampling for preparation of the analytical samples shall proceed in accordance with ISO 14284.

The distribution of oxygen within the product is dependent on the manufacturing process. The result of the oxygen determination will therefore depend on the sample position. This has to be agreed if it is not specified in the product standard.

7 Sample preparation

7.1 Method A Punching

7.1.1 Saw off slices 3 mm to 4 mm thick from the sample piece using a mechanical saw (5.1.1). The number shall be as required by the method.

NOTE: Figures 3 and 4 give examples of the different procedures for different sample shapes.

7.1.2 Use a flat bed linisher (5.2.2) with 60 grade silicon carbide paper so that the sample slices are ground flat on both sides removing most of the oxide layer. Hold the sample with a magnetic holding device during grinding.

7.1.3 Remove thoroughly any oxides remaining on the surface with a rotary file under a flow of argon or nitrogen (5.2.3). The condition of the surface after preparation shall be smooth, bright and free from burrs and irregularities. Avoid significant heating of the sample in order to prevent oxidation of the surface after removal from inert atmosphere.

After treatment, handle the samples with tweezers only. Do not touch the samples by hand.

NOTE: During milling and all subsequent preparations, take care to ensure that the tools and objects coming into contact with the sample are free from oxygen containing contaminants.

7.1.4 The test samples should usually have a mass of approximately 1 g. The mass of the sample depends on the method of oxygen determination. Prepare the samples with a punch (5.2.1). A punch of 4 to 6 mm diameter is normally suitable for this purpose. During the punching operation purge the receptacle into which the sample is collected, with argon or nitrogen (4.2) (figure 1) Keep the test samples in a glass receptacle (5.1.3). Samples should be analysed as soon as possible on the same day as preparation.

7.1.5 Immediately before analysis, wash the test samples in organic solvent (4.1) and dry them in air or under vacuum free from backgassing.

NOTE: Problems with microcracks have been encountered in the determination of low levels of carbon (< 10ppm). There may be similar contamination problems in the determination of oxygen.

7.2 Method B Turning

7.2.1 Take rectangular pieces about 100 mm long with a cross section of about 10 mm x 10 mm from the sample piece with a mechanical saw. Figures 3 and 4 give examples of the various procedures for different shapes of samples.

If rods, wire or forged pieces of about 10 mm diameter are available, it is only necessary to cut off one piece about 100 mm in length.

7.2.2 The test samples should usually have a mass of approximately 1 g. The mass of the sample depends on the method of oxygen determination. Prepare these samples by turning the pieces on a lathe (5.3.1). For this purpose, turn down the sample obtained in accordance with 7.2.1, to about 7 mm diameter at a speed of about 1000 rev/min. Subsequent turning down to 6 mm diameter shall be at a speed of 250 rev/min with a feed of about 0,2 mm/rev. After turning, file the surface with a fine file (5.3.4) with the lathe still running until the sample is smooth and bright. Avoid significant heating of the sample in order to prevent oxidation of the surface.

As an alternative to slow-speed turning, reduce the diameter from 7 mm to 6 mm using a speed of between 800 rev/min to 1000 rev/min and a feed of between 0,1-0,15 mm/rev. In this case, the cylindrical surface is smooth enough to dispense with the filing operation.

NOTE: Use no cooling lubricant during turning down from 7 mm to 6 mm. Take care also during the subsequent stages in preparation to ensure that any objects coming into contact with the sample are free from oxygen containing contaminants.

7.2.3 Using the hand saw (5.3.2), saw off the end of the sample which is not used for analysis. Clean the accessible end face of the clamped sample by filing. Then, saw off a test sample 4 mm to 5 mm long for analysis.

Handle the sample with tweezers only. Do not touch samples by hand. Use the tweezers for all manipulations. File carefully the unfiled end face of the sample in a filing block (figure 2) or in another similar holding device. Remove carefully any adhering dust with a small brush (5.3.5). Keep the test samples in a glass receptacle (5.1.3). These shall be analysed as soon as possible on the same day as they were prepared.

Immediately before analysis, wash the samples in organic solvent (4.1) and dry them in air or under vacuum free from backgassing.

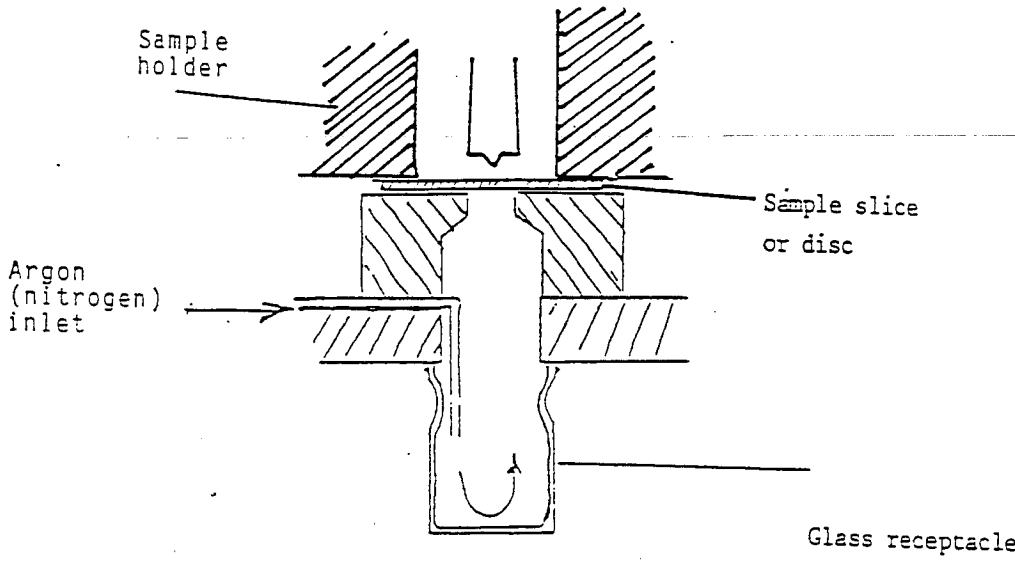

Figure 1 - Punch for making samples for oxygen analysis