SLOVENSKI STANDARD
oSIST prEN ISO 643:2023

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Nadomešča:

SIST EN ISO 643:2020

Jekla - Mikrografsko določevanje navidezne velikosti kristalnih zrn (ISO/DIS 643:2023)

Steels - Micrographic determination of the apparent grain size (ISO/DIS 643:2023)

Stahl - Mikrophotographische Bestimmung der erkennbaren Korngröße (ISO/DIS 643:2023)

Aciers - Détermination micrographique de la grosseur de grain apparente (ISO/DIS 643:2023)

Ta slovenski standard je istoveten z: **prEN ISO 643**

ICS:

77.040.99	Druge metode za preskušanje kovin	Other methods of testing of metals
77.080.20	Jekla	Steels

oSIST prEN ISO 643:2023 **en,fr,de**

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DRAFT INTERNATIONAL STANDARD
ISO/DIS 643

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Steels — Micrographic determination of the apparent grain size

Aciers — Détermination micrographique de la grosseur de grain apparente

ICS: 77.040.99

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This fifth edition cancels and replaces the fourth edition (ISO 643:2019), which has been technically revised.

The main changes are as follows:

— the test temperature of McQuaid-Ehn method has been increased to 950 °C (see 6.3.4);

— subclause 7.1.2 has been modified with reference to new Annex B and amended Table 2;

— Annex B from the third edition (ISO 643:2012) has been reinstated, now with new ISO grain size charts instead of ASTM charts;

— the old Annex B (evaluation method) has been renumbered as Annex C. New Formulas (C.8) and (C.9) and amended Table C.1 have been introduced.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steels — Micrographic determination of the apparent grain size

WARNING — This document calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This document does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this document to establish appropriate health, safety and environmentally acceptable practices.

1 Scope

This document specifies a micrographic method of determining apparent ferritic or austenitic grain size in steels. It describes the methods of revealing grain boundaries and of estimating the mean grain size of specimens with unimodal size distribution. Although grains are three-dimensional in shape, the metallographic sectioning plane can cut through a grain at any point from a grain corner, to the maximum diameter of the grain, thus producing a range of apparent grain sizes on the two-dimensional plane, even in a sample with a perfectly consistent grain size.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Grains

3.1.1
grain
closed polygonal shape with more or less curved sides, which can be revealed on a flat section through the sample, polished and prepared for micrographic examination

Note 1 to entry: In ISO 4885 grain is defined as "space lattice formed by atoms with regular interstices".

3.1.2
austenitic grain
crystal with a face-centred cubic crystal structure which may, or may not, contain annealing twins

3.1.3
ferritic grain
crystal with a body-centred cubic crystal structure which never contains annealing twins

Note 1 to entry: If pearlite colonies of similar dimensions to those of the ferrite grains are present, the colonies are then counted as ferrite grains.

3.2   General

3.2.1
index
positive, zero or possibly negative number G which is derived from the mean number m of *grains* ([3.1.1](#)) counted in an area of 1 mm² of the section of the specimen

Note 1 to entry: By definition, $G = 1$ where $m = 16$; the other indices are obtained by [Formula (1)](#).

3.2.2
intercept
N
number of *grains* ([3.1.1](#)) intercepted by a test line, either straight or curved

Note 1 to entry: See [Figure 1](#).

Note 2 to entry: Straight test lines will normally end within a grain. These end segments are counted as 1/2 an intercept. $\bar{N}$ is the average of a number of counts of the number of grains intercepted by the test line applied randomly at various locations. $\bar{N}$ is divided by the true line length, L_T usually measured in millimetres, in order to obtain the number of grains intercepted per unit length, $\bar{N}_L$.

3.2.3
intersection
P
number of intersection points between *grain* ([3.1.1](#)) boundaries and a test line, either straight or curved

Note 1 to entry: See [Figure 2](#).

Note 2 to entry: $\bar{P}$ is the average of a number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations. $\bar{P}$ is divided by the true line length, L_T usually measured in millimetres, in order to obtain the number of grain boundary intersections per unit length, $\bar{P}_L$.

4   Symbols

The symbols used are given in [Table 1](#).

Table 1 — Symbols

Symbols	Definition	Value
$\bar{a}$	Mean area of grain in square millimetres	$\bar{a} = \dfrac{1}{m}$
A_F	Apparent area of the test figure in square millimetres	—
$\bar{d}$	Mean grain diameter in millimetres	$\bar{d} = \dfrac{1}{\sqrt{m}}$
D	Diameter of the circle on the ground glass screen of the microscope or on a photomicrograph enclosing the image of the reference surface of the specimen	79,8 mm (area = 5 000 mm²)
g	Linear magnification (to be noted as a reference) of the microscopic image	In principle 100
G	Equivalent index of grain size	$G = \log_2 m - 3$
K	Conversion factor from linear magnification × g to linear magnification ×100	$K = \dfrac{g}{100}$
l	Mean lineal intercept length, generally expressed in millimetres	$l = 1 / \bar{N}_L = 1 / \bar{P}_L$
l_0	Mean lineal intercept length for G = 0, in millimetres	0,313
a	The method for designating the direction conforms to ISO 3785.	

Table 1 *(continued)*

Symbols	Definition	Value
L_T	True length of the test line divided by the magnification, in millimetres	—
m	Number of grains per square millimetre of specimen surface in the area examined	$m = 2\,n_{100}$ (magnification × 100) $m = 2\,K^2 n_g$ (magnification × g)
M	Number of the closest standard chart picture where g is not 100	—
n_g	Total equivalent number of grains examined on the image of diameter D (with a magnification × g)	—
n_1	Number of grains completely inside the circle of diameter D	—
n_2	Number of grains intersected by the circle of diameter D	—
n_{100}	Total equivalent number of grains examined on the image of diameter D (with magnification × 100)	—
$\overline{N}$	Mean number of grains intercepted per unit length L	—
$\overline{N}_L$	Mean number of grains intercepted per unit length of the line	$\overline{N_L} = \overline{N} / L_T$
N_x	Number of intercepts per millimetre in the longitudinal direction[a]	—
N_y	Number of intercepts per millimetre in the transverse direction[a]	—
N_z	Number of intercepts per millimetre in the perpendicular direction[a]	—
$\overline{P}$	Mean number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations	—
$\overline{P}_L$	Mean number of grain boundary intersections per unit length of test line	$\overline{P_L} = \overline{P} / L_T$
Q	Correction factor for non-standard magnification	$Q = 2\,log_2\left(\dfrac{g}{100}\right)$

[a] The method for designating the direction conforms to ISO 3785.

5 Principle

This document is applicable to grain structures that have a normal (Gaussian) size distribution. The apparent grain size is determined by micrographic examination of appropriately prepared sections of the specimen.

The following principal methods are available to obtain an index representing the mean value of the grain size:

a) comparison method using standard charts (see clause 7.2);

b) planimetric method counting grains to determine the mean number of grains per unit area, (see clause 7.3);

c) intercept method counting the number of grains or grain boundaries along a line of a known length (see clause 7.4).

All methods give comparable results.

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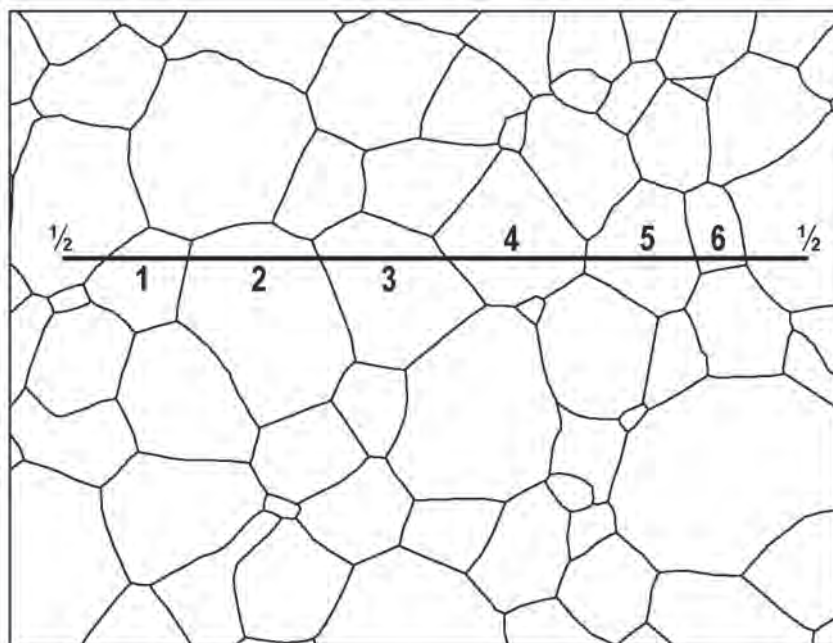
NOTE   Intercept, N, counts for a straight line on a single-phase grain structure where the arrows point to 6 intercepts and two line segments ending within grain ($2 \times 0,5 = 1\ N$) and $N = 7$.

Figure 1 — Example of intercept, N
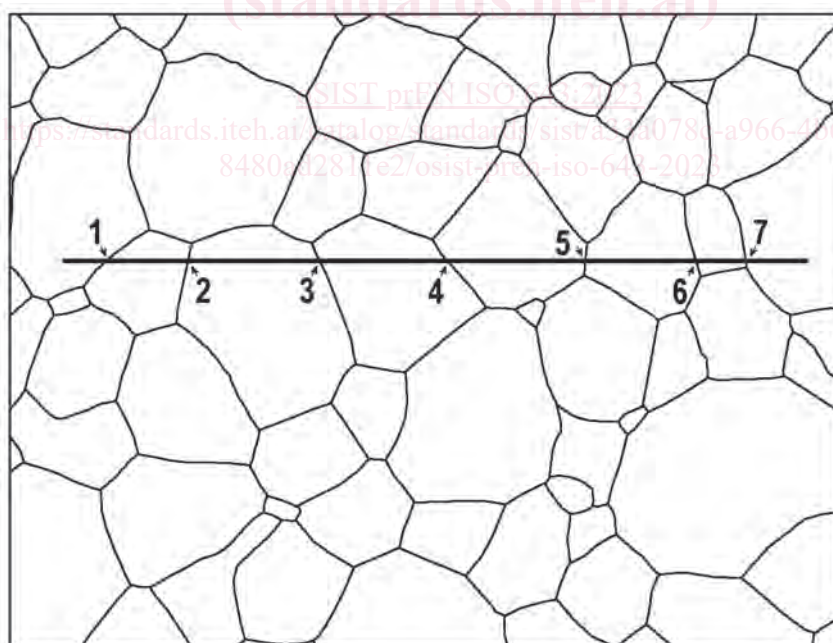
NOTE   Intersection, P, counts for a straight test line placed over a single-phase grain structure where the arrows point to 7 intersection points and $P = 7$.

Figure 2 — Example of intersection, P

6 Selection and preparation of the specimen

6.1 Test location

If the order, or the standard defining the product, does not specify the number of specimens and the point at which they are to be taken from the product, these are left to the manufacturer, although it has been shown that precision of grain size determination increases the higher the number of specimens assessed. Care shall be taken to ensure that the specimens are representative of the bulk of the product (i.e. avoid heavily deformed material such as that found at the extreme end of certain products or where shearing has been used to remove the specimen, etc.). The specimens shall be polished in accordance with the usual methods.

Unless otherwise stated by the product standard or by agreement with the customer, the polished surface can be randomly selected for the specimens with equiaxial grains. The polished surface shall be parallel to the principal axis of deformation in wrought products, for the specimens with deformed grains.

NOTE   Measurements of the grain size on a transverse plane will be biased if the grain shape is not equiaxial.

6.2 Revealing ferritic grain boundaries

The ferritic grains shall be revealed by etching with nital (ethanolic 2 % to 3 % nitric acid solution), or with another appropriate reagent.

NOTE   In this standard, all chemical compositions are indicated in mass %.

6.3 Revealing austenitic and prior-austenitic grain boundaries

6.3.1 General

In the case of steels having a single-phase or dual-phase mainly austenitic structure (delta ferrite grains in an austenitic matrix) at ambient temperature, the grains shall be revealed by an etching solution. For single phase austenitic stainless steels, the most commonly used chemical etchants are glyceregia, Kalling's reagent (No. 2) and Marble's reagent. The best electrolytic etch for single or two-phase stainless steels is aqueous 60 % nitric acid at 1,4 V d.c. for 60 s to 120 s, as it reveals the grain boundaries but not the twin boundaries. Aqueous 10 % oxalic acid, 6 V d.c., up to 60 s, is commonly used but is less effective than electrolytic 60 % nitric acid.

For other steels, one or other of the methods specified below shall be used depending on the information required.

— "Bechet-Beaujard" method by etching with aqueous saturated picric acid solution (see A.2).

— "Kohn" method by controlled oxidation (see A.3).

— "McQuaid-Ehn" method by carburization (see A.4).

— grain boundary sensitization method (see A.7).

— other methods specially agreed upon when ordering.

NOTE   The first three methods are for prior-austenitic grain boundaries while the others are for austenitic Mn or austenitic stainless, see Annex A.

If comparative tests are carried out for the different methods, it is essential to use the same heat treatment conditions. Results may vary considerably from one method to the other.

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7 Characterization of grain size

7.1 General

7.1.1 Characterization methods

The apparent grain size can be determined by three micrographic methods: comparison method, planimetric method and intercept method.

7.1.2 Formulae

The index is defined by Formula (1):

$$m = 8 \times 2^G \tag{1}$$

This formula may be stated as Formula (2):

$$G = \log_2 m - 3 \tag{2}$$

NOTE   An alternative system of grain size definition is known as the ASTM grain size (see C.2).

7.1.3 Measurement uncertainty

In general, the comparison method allows for an accuracy of 0,5; the planimetric and intercept segment methods allow for an accuracy of 0,1, see Bibliography [4]. For comparison between methods, the indexes obtained are usually rounded to multiples of 0,5.

Due to the randomness of the spatial position in which each grain is cut through by the sectioning plane and due to the measurement error, no determination of apparent grain size can be an exact measurement. Therefore, for planimetric and intercept methods it can be of interest to calculate the 95 % confidence interval of the grain size measurement result and adjust the number of fields inspected according to the percentage relative accuracy, % RA, of counting corresponding to the uncertainty of ±0,25 grain size units, taking into account that for a symmetric error of G the % RA of the measured quantity is not symmetric, see Annex D.

The methods described in this standard yield representative results for specimens with a unimodal grain size distribution. Applying them to specimens with bimodal (or more complex) size distributions will yield an average value that likely has no meaningful relationship with the various grain populations, but may still represent the specimen on average. ISO 14520 may be the more appropriate standard for characterizing these specimens, see Annex E.

7.2 Comparison method

7.2.1   The image examined on the screen (or on a photomicrograph) shall be compared with a series of standard charts presented in Annex B or overlays (eye-piece graticules designed for grain size measurement can be used providing these are traceable to national or international standards). The standard charts at a magnification of × 100 are numbered from 00 to 10 so that their number is equal to the index G. Using ASTM E 112 images gives substantially the same results as using the comparison charts of Annex B.

NOTE   All standard charts in Annex B are displayed at a magnification of ×100. The different sizes of circles are used between 00 to 2,5 (circle of 2 mm^2) and 3,0 to 10 (circle of 0,5 mm^2).

7.2.2   The standard chart with the grain size closest to that of the examined fields of the specimen can then be determined. A minimum of three randomly selected fields shall be assessed on each specimen.

7.2.3 Where the magnification g of the image on the screen or photomicrograph is not ×100, the index G shall be equal to the number M of the closest standard chart, modified as a function of the ratio of the magnifications, as given by Formula (3):

$$G = M + 2\log_2 \frac{g}{100} \tag{3}$$

7.2.4 Table 2 gives the relationship between the indices for the usual magnifications.

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Table 2 — Relationship between indices for the usual magnifications

Standard chart no. M	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10
Magnification of the image g / Q^a									Grain size										
25 / −4	−3	−2,5	−2	−1,5	−1	−0,5	0	0,5	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6
50 / −2	−1	−0,5	0	0,5	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8
100 / 0	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10
200 / +2	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12
400 / +4	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14
500 / +4,5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5
800 / +6	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5	15	15,5	16
1 000 / +6,5	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5	15	15,5	16	16,5

[a] The values for g = 500 and g = 1 000 are rounded to the nearest multiple of 0,5.

7.2.5   For the comparison method, if the difference between the maximum index and the minimum index determined is less than 3, compute the test result as the arithmetic mean of the found indices. If the indices are calculated using Formula (4), the arithmetic mean is to be calculated after the modification for non-standard magnification. If this condition is not fulfilled, perform an additional series of at least 6 determinations of the grain size. If the difference between the maximum index and the minimum index determined in this new series is less than 3, then compute the test result as the arithmetic mean of the first and second series of determinations altogether. If this latter condition is not fulfilled, note the spread and a comment on the findings in the final report. For a more elaborate discussion on specimens of non-unimodal distribution, see Annex E.

The calculated arithmetic mean of indices shall be rounded to the nearest multiple of 0,5.

7.3   Planimetric method

7.3.1   Historically a circle measuring 79,8 mm in diameter (A_F = 5 000 mm^2), see Figure 3, was drawn or superimposed over a micrograph or a live image on a ground glass projection screen. The magnification was then adjusted so that the circular area contained at least 50 grains in order to minimize the counting error associated with a circular test pattern. The following procedure and formulae are magnification neutral.

NOTE   The circle referenced an apparent size specifically at ×100 magnification as perceived by an operator at a microscope using ×10 oculars and ×10 objective. This reference also was, and still is, used in other applications such as inclusion assessments. It is moreover a component of the recommended concentric circle grid used in the intercept method, as well as one of the reference circles used in the comparison method.

However, more recent tools like image software allow for optimizing the combination of circle diameter and magnification to facilitate the count and to make sure that the number of grains within the circle reach at least 50. Therefore, it is no longer always relevant to reference a specific circle size at a specific magnification.