INTERNATIONAL
STANDARD

ISO
377-2

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Selection and preparation of samples and test pieces of wrought steels —

Part 2:
Samples for the determination of the chemical composition

Prélèvement et préparation des échantillons et éprouvettes en aciers corroyés —
Partie 2: Échantillons pour la détermination de la composition chimique

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Reference number
ISO 377-2 : 1989 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 377-2 was prepared by Technical Committee ISO/TC 17, *Steel.*

Together with International Standard ISO 377-1, it cancels and replaces the first edition of ISO 377 (ISO 377 : 1985), of which it constitutes a technical revision.

ISO 377 consists of the following parts, under the general title *Selection and preparation of samples and test pieces of wrought steels*:

- *Part 1: Samples and test pieces for mechanical test*
- *Part 2: Samples for the determination of the chemical composition*

Annex A of this part of ISO 377 is for information only.

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Selection and preparation of samples and test pieces of wrought steels —

Part 2:
Samples for the determination of the chemical composition

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1 Scope

1.1 This part of ISO 377 specifies all the operations by which samples intended for the determination of the chemical composition are obtained from a given product. The determination of hydrogen is excluded.

These samples are for use in tests which, unless agreed to the contrary when ordering, are carried out in conformity with the methods indicated in the product standard or, in the absence of this, in the test standard. For the purpose of comparison with a specification, the sample represents the average composition.

There are two groups of methods for the determination of the chemical composition:

a) methods in which the sample is subjected to a chemical reaction; these are designated by the name "chemical methods" (see clause 5);

b) methods in which the determination of the constituent elements is carried out without a chemical reaction; these are designated by the name "physical methods" (see clause 6).

1.2 This part of ISO 377 applies to crude products[1], semi-finished products and finished wrought steel products, dealt with in ISO 404 and does not apply to samples during the melting or refining stage of production. In cases where the product standards or the test standards specify different conditions, these different conditions are applicable.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 377. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 377 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-1 : 1989, *Selection and preparation of samples and test pieces of wrought steels — Part 1: Samples and test pieces for mechanical test.*

ISO 404 : 1981, *Steel and steel products — General technical delivery requirements.*

3 Definitions

NOTE — The equivalent English, French and Russian terms are given in annex A. They are illustrated in figure A.1.

For the purposes of this part of ISO 377, the following definitions apply.

3.1 sample product: Item (a sheet, for example) selected from a supplied batch for the purpose of obtaining test portions.

3.2 sample: Quantity of solid material removed from the sample product for the purpose of taking samples for analysis (see 3.3).

3.3 sample for analysis: Quantity of material taken from the sample, or directly from the product, in such a way as to represent the mean chemical composition of the product.

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[1] In the context of this standard, the concept of "crude products" includes not only ingots, but also continuously cast products intended for subsequent transformation such as blooms, billets, slabs and all products with other sections.

The following categories are distinguished:

- sample in the form of chips (see clause 5);
- solid samples (see 6.1);
- compacted samples (see 6.3);
- remelted samples (see 6.4).

3.4 test portion: Part of the sample for analysis, or the sample itself which is submitted to the analysis.

4 Selection and preparation of samples

4.1 Location and dimensions of samples

The samples shall be selected at the location specified by the product standard.

In the absence of requirements given in the product standard, or of a specification when ordering, the samples for analysis shall be selected from the test piece or samples for mechanical testing (see ISO 377-1, 5.1) or directly from the product. In the latter case, for rolled products, the selection process shall be carried out on a section or semi-section at right angles to the principal direction of working and at one end of the product.

The dimensions of the samples shall be sufficient to permit any re-tests which may be necessary.

4.2 Identification of samples

Samples shall be provided with the identification marks which are required or considered necessary in order to determine the product from which they were taken and their location in the product.

4.3 Preparation of the surface of the sample

If present, surface coating or contaminants, such as oxide, paint or grease, shall be removed by any suitable means and, if necessary, degreased using a suitable solvent.

In the case of products susceptible to surface changes in composition, it may be agreed to remove from the sample the parts corresponding to the surface area which has changed.

Following these operations, the sample shall be protected from contamination.

4.4 Conservation of samples for analysis

The samples shall be conserved in such a way as to prevent any contamination or change, in completely dry containers made from a chemically inert material highly resistant to abrasion by steel.

The containers shall be provided with the necessary marks and sealed if necessary. Containers intended for any arbitration shall be sealed by the manufacturer and by the users or by their representatives.

Unless agreed to the contrary, these containers shall be kept by the party responsible for the selection of samples.

5 Selection and preparation of samples for analysis in the form of chips for the determination of the chemical composition by chemical methods of analysis

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5.1 Machining or cutting

The tools, machines and containers used during the preparation of the samples shall be cleaned beforehand to prevent any contamination of the sample for analysis.

If sampling applies to the whole of the cross-section, planing, milling, turning or cutting shall be used.

If sampling is carried out on one part of the cross-section only, or at specified locations, drilling or one of the previous processes shall be used according to the case.

The use of lubricants and coolants is only permitted in exceptional cases. In this case the chips shall be cleaned by means of suitable solvents which do not leave any deposits.

Machining shall be carried out in such a way that the chips are not oxidized by overheating. Unavoidable coloration of chips from some manganese and austenitic steels which become hot during machining shall be minimized by selection of appropriate tools and cutting speeds.

Sampling shall be carried out in such a way as to obtain sufficiently thin chips to minimize any subsequent fragmentation process required in the preparation of the sample for analysis (see 5.2.1, second paragraph).

Generally the size of the chips shall be such that they are between 2,5 mg and 25 mg in mass per chip.

For unalloyed and low-alloy steels, the chips shall be approximately 10 mg and, for high-alloy steels, approximately 2,5 mg.

In the case of graphitic steel, the chips shall be as solid and compact as possible (approximately 100 mg) to prevent crumbling or loss of the graphite. Chips taken by machining are not suitable for the determination of oxygen.

5.2 Fragmentation and sieving

5.2.1 Fragmentation

If the number of chips obtained in accordance with 5.1 is insufficient, fragmentation shall be carried out using a suitable crusher. As in the case of machining, the crusher shall be cleaned beforehand.

If the fragmentation process cannot be applied, an appropriate cutting process shall be used.

5.2.2 Sieving

If sieving is necessary to obtain a representative sample, the whole of the sample shall be passed through a sieve with a mesh opening of 2,5 mm.

If necessary, oversized particles shall be broken up until sieving is complete.

If the sample contains dust, i.e. chips that pass through a sieve with a mesh opening of 0,050 mm, this dust shall be separated by sieving and the two fractions thus obtained weighed. The test portion shall be made up of two parts proportional to these fractions.

For the determination of carbon, the whole of the sample shall be passed through a sieve with a mesh opening of 2,5 mm.

For the determination of nitrogen, the sample shall be machined so that the number of individual fragments less than 0,050 mm is as low as possible.

The sieving operations shall be carried out taking all the precautions necessary to avoid contamination, changes or losses of material.

5.3 Mass of the sample for analysis

A sample for analysis shall be prepared which is of sufficient mass to provide for any necessary re-analyses. The quantity of material sampled shall be obtained by weighing. Generally, a mass of approximately 100 g is sufficient.

5.4 Conservation of samples for analysis

The requirements of 4.4 shall apply.

It is permitted to conserve samples for analysis as a solid mass, and chips may then be prepared when required.

6 Selection and preparation of solid, compacted or remelted samples for determination of the chemical composition by physical methods of analysis

(Emission spectrometry in the visible and ultra-violet regions of the spectrum, X-ray fluorescence, mass spectrometry, etc.).

Depending on the requirements of the product standard or any agreement between the parties, either

a) a solid sample, or

b) a compacted or remelted sample shall be used.

NOTE — Not all grades of steel can be compacted or remelted.

The part actually analysed consists of a small volume of the sample for analysis. This volume shall be compatible with the method of analysis.

6.1 Solid sample

The solid sample shall be obtained by cutting from the sample a piece which may be placed on the sample table or placed in the sample holder of the analytical apparatus.

Cutting shall be carried out by sawing, abrasive cutting, section cutting, shearing or stamping.

The surface shall be machined by grinding, by milling or with abrasive paper to obtain the surface quality required for the method of analysis used. For the determination of silicon by X-ray fluorescence spectroscopy or emission spectrometric analysis, an alumina abrasive shall be used. For the determination of aluminium by X-ray fluorescence spectroscopy or emission spectrometric analysis, a silicon abrasive shall be used. For the determination of carbon by emission spectrometric analysis, an oxide-based abrasive shall be used.

In the absence of any indication in the product standard, the analysis shall be carried out on the part of the sample corresponding to a transverse section of the product, if the sample has sufficient thickness.

6.2 Solid sample with a thickness of less than 1,5 mm

Physical methods in which an arc or a spark is produced between the solid sample and a counter electrode (emission spectrometry in the visible and ultra-violet regions of the spectrum or mass spectrometry) cause the solid sample to heat up. The thinner the solid sample, the greater the local heating.

For solid samples with a thickness of less than 1,5 mm, it may be necessary to adopt special techniques to reduce the local heating during sparking. For example, the edges of the solid sample may be electrically welded1) to a small block of steel or the solid sample may be bedded in tin, leaving one surface bare.

A surfacing operation shall then be carried out as described in 6.1.

6.3 Compacted sample

The preparation of chips shall be carried out by the same methods and under the same conditions as for the samples intended for analysis by chemical methods (see 5.1 and 5.2).

About 10 g of fine chips of regular shape shall be placed in a metal ring with an inside diameter of approximately 25 mm. This ring shall be placed on a solid steel base, the upper surface

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1) Using the autogenous Tungsten Inert Gas (TIG) welding process.

of which is perfectly flat. Using a press and a piston which fits easily into the ring with only a little friction, compression shall be carried out under a load of at least 1 800 MPa[1].

The surface of the compacted sample-ring assembly shall then be treated as described in 6.1 and subjected as such to analysis by physical methods.

The quality of the surface submitted to the analysis depends, to some extent, on the dimensions and shape of the chips. The use of very fine chips which can pass through a sieve with a mesh opening of less than 0,80 mm shall be avoided.

The calibration curves of the analytical apparatus shall be drawn up from the results obtained using compacted samples.

6.4 Remelted sample

Chips, solid samples or small pieces of steel may be remelted in an inert atmosphere in special melting equipment, such as a high-frequency or argon-arc furnace, to convert them to a form suitable for spectral analysis.

It is essential to ensure that any chemical change which occurs during remelting is quantitatively known and that it does not significantly influence the test result.

NOTE — To minimize any changes in the chemical composition during remelting, frequently 0,05 g of zirconium are added as a deoxidant and the instrument is calibrated using remelted standard samples.

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[1] 1 MPa = 1 N/mm^2 = 10 bar

Annex A
(informative)

Glossary of terms relating to samples for the determination of chemical composition

English	French	Russian	Sub-clause
Sample product	Produit échantillon	Образец-продукт	3.1
Sample	Échantillon	Образец	3.2
Sample for analysis	Échantillon pour analyse	Образец для анализа	3.3
Test portion	Prise d'essai	Взвешенный и отборенный образец	3.4
Solid sample	Échantillon massif	Твердый образец	6.1
Compacted sample	Échantillon compacté	Сжатый образец	6.3
Remelted sample	Échantillon refondu	Переплавленный образец	6.4

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