
**Welding and allied processes —
Determination of hydrogen content in
arc weld metal**

*Soudage et techniques connexes — Détermination de la teneur en
hydrogène dans le métal fondu pour le soudage à l'arc*

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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 on 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 the following URL: www.iso.org/iso/foreword.html (standards.iteh.ai)

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Any feedback, question or request for official interpretation related to any aspect of this document should be directed to IIW via your national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

This fourth edition cancels and replaces the third edition (ISO 3690:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

- an additional specimen size D has been added;
- changes have been made in required diffusion times for high temperature tests, see [5.3.3.4](#), [5.3.4](#) and [Table 5](#).

Welding and allied processes — Determination of hydrogen content in arc weld metal

1 Scope

This document specifies the sampling and analytical procedure for the determination of diffusible hydrogen in martensitic, bainitic, and ferritic steel weld metal arising from the welding of such steels using arc welding processes with filler material.

The techniques specified in this document include collection of diffusible hydrogen via displacement of mercury or collection into a headspace filled with an inert gas such as argon. The amount of hydrogen collected is determined by measuring the displaced volume in the former and by, for example, thermal conductivity in the latter.

The temperature for collection of diffusible hydrogen is controlled to avoid thermal activation of non-diffusible hydrogen.

NOTE Recommendations and restrictions in regard to older methods of measurement using glycerine are given in [Annex B](#) for any comparison work to these older methods.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14175, *Welding consumables — Gases and gas mixtures for fusion welding and allied processes*

ISO/TR 17671-1, *Welding — Recommendations for welding of metallic materials — Part 1: General guidance for arc welding*

ISO 80000-1:2009, *Quantities and units — Part 1: General*

3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

Filler material is deposited on to a standard test coupon in a manner that ensures control of pertinent variables to produce a representative specimen for analysis. Subsequent storage and handling of the specimen is controlled to prevent premature loss of hydrogen. Finally, the specimen is transferred to a gas collection apparatus (mercury method) or to a suitable vessel filled with an inert gas (thermal conductivity method) and held for a period of time at a temperature sufficient to quantitatively release the diffusible hydrogen into an evacuated gas burette or into the inert gas headspace, respectively. The amount of hydrogen collected is determined by measuring the displaced volume (mercury method) or by thermal conductivity. Finally, quantification of the mass of deposited metal or volume of fused weld

metal enables calculations of diffusible hydrogen in deposited metal, H_D , or diffusible hydrogen in fused weld metal, H_F , to be made.

NOTE [Annex C](#) gives information on determination of accuracy of results when a method other than displacement of mercury or thermal conductivity detection is used for diffusible hydrogen analysis.

5 Test procedures

5.1 Production of weld specimens

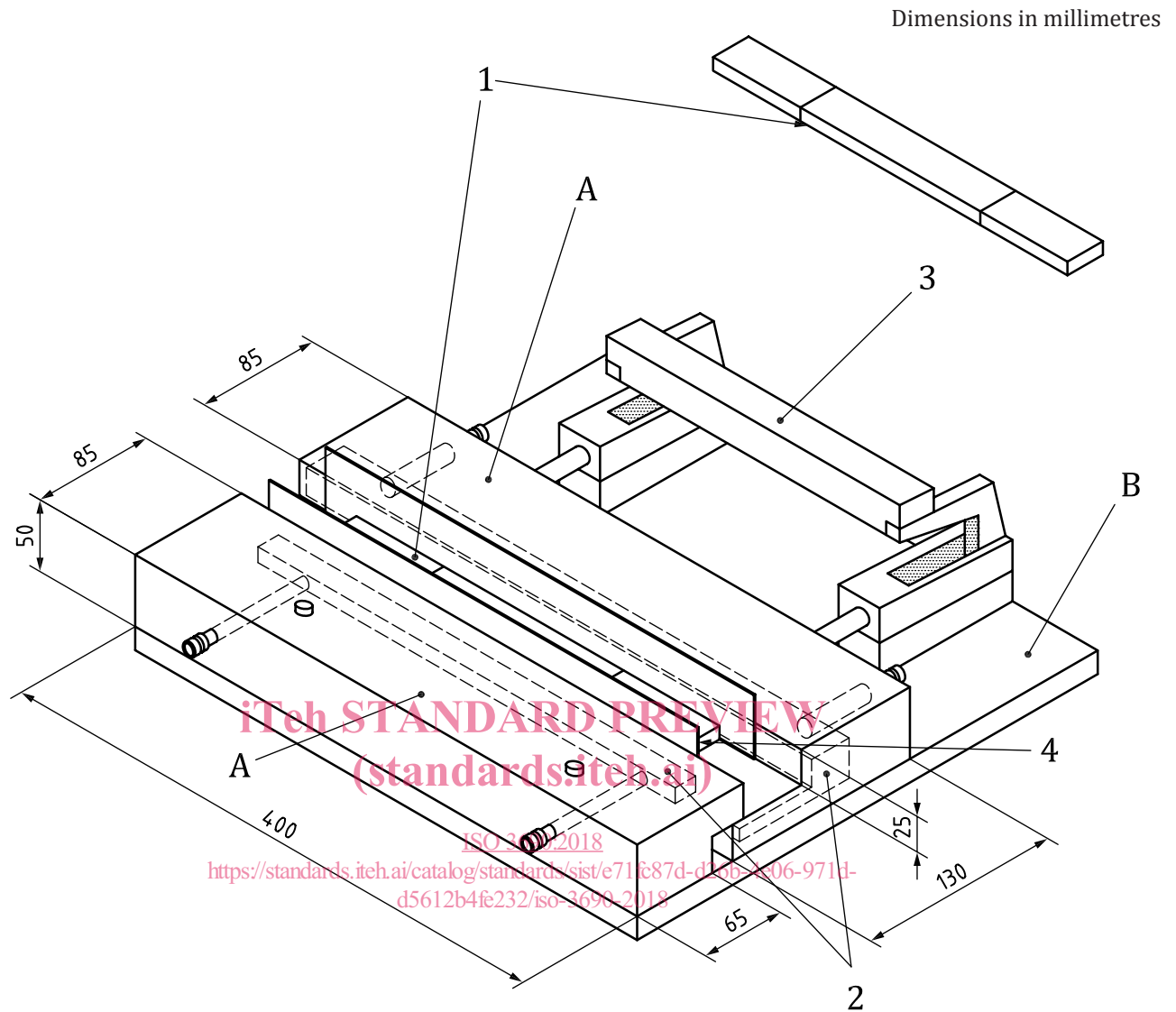
5.1.1 Summary

The welding consumable to be tested is used to deposit a single weld bead, which is rapidly quenched and subsequently stored at -78 °C or lower until analysis. Cleaning and slag removal are performed on the chilled specimen.

5.1.2 Welding fixture

An example of a suitable welding fixture to provide uniform test pieces for the welding processes specified in [5.2](#) is shown in [Figure 1](#). It is designed to hold the uniform test pieces securely in alignment during welding and, in particular, to ensure that unclamping upon completion of welding can be carried out in a single operation according to the conditions specified in [5.1.4 c\)](#). The surface temperature of the fixture shall be between ambient and 25 °C above ambient at the start of each test weld. The fixture may be water-cooled to decrease the cycle time. The temperature of the cooling water shall be controlled to prevent condensation of water on the surface of the fixture between test welds.

For all welding processes, the test piece assembly is clamped in the welding fixture using annealed copper foil as shown in [Figure 1](#). The foil may be annealed repeatedly and quenched in water after each annealing. Oxide scale after annealing is removed by pickling with dilute nitric acid (10 % by volume) followed by washing with distilled water and drying.



Key

- 1 test piece assembly per [Figure 2](#)
- 2 water cooling jacket (if necessary)
- 3 lever clamp
- 4 copper foil inserts (1 mm × 15 mm minimum × 300 mm)
- A copper
- B carbon steel

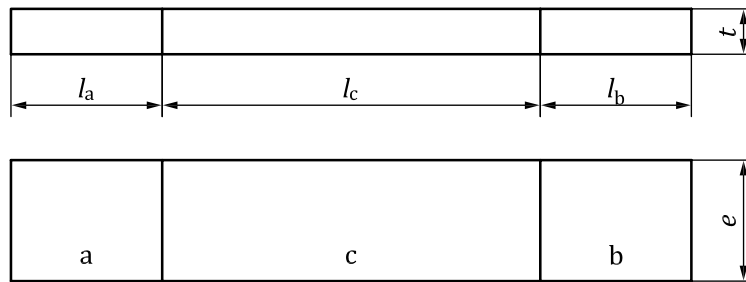
Figure 1 — Example of a suitable welding fixture and test piece assembly for weld deposits

5.1.3 Test piece assemblies

The test piece assembly shall be prepared from plain carbon non rimming steel with a carbon content of not more than 0,18 % by mass and a sulfur content of not more than 0,02 % by mass. The assembly shall conform to the dimensions shown in [Figure 2](#) and [Table 1](#) for assembly A, assembly B, assembly C, or assembly D with a tolerance of $\pm 0,25$ mm on all dimensions except the length of the run on and run off pieces. The lengths shown in [Figure 2](#) and [Table 1](#) for the run on and the run off piece represent minimum values.

All surfaces shall be finished at right angles to ensure good contact between adjacent pieces during the welding operation. Each test piece assembly may be finished with one operation on a surface grinder so as to ensure a uniform width, or closer dimensional control may be exercised to obtain proper clamping. See 5.1.4 d) for evidence of proper clamping.

The centre test piece shall be numbered by engraving or stamping on the opposite side of that used for welding. The entire test piece assembly shall be degassed at $650\text{ °C} \pm 10\text{ °C}$ for 1 h and cooled in either a dry inert gas atmosphere or a vacuum. Alternatively, the test piece assembly may be degassed and cooled in air if the surface oxide layer is removed prior to testing. Degassed test piece assemblies shall be stored in a desiccator or under other suitable conditions to prevent oxidation of the test pieces. After numbering and removal of oxide, the mass, m_1 , of each centre test piece shall be determined to the nearest 0,1 g for assembly A or to the nearest 0,01 g for assembly B, assembly C, or assembly D.



Key

- a run on piece of length l_a
- b run off piece of length l_b
- c centre test piece of length l_c
- e test piece width
- t test piece thickness

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Figure 2 — Dimensions of the weld test assembly

Table 1 — Dimensions of the weld test assembly

Dimensions in millimetres

Test assembly	l_a and l_b	l_c	e	t
A ^a	≥25 (50)	80	25	12
B	≥25 (50)	30	15	10
C	≥50	15	30	10
D	≥25 (50)	40	25	12
$l_a \geq 25\text{ mm}$ and $l_b \geq 25\text{ mm}$: manual metal arc welding				
$l_a \geq 50\text{ mm}$ and $l_b \geq 50\text{ mm}$: other welding processes				
^a Identical to the specimen geometry according to AWS A4.3-93.				

5.1.4 Welding and test piece storage

The temperature of the welding fixture before each weld is made shall be ambient or not more than 25 °C above ambient. If difficulty is caused by condensation of water on the fixture and test piece assembly, it is necessary to use cooling water thermostatically controlled to ambient temperature or as much as 25 °C higher. Using the welding process as specified in 5.2, and parameters appropriate to the type of investigation, a single weld bead shall be made on the test piece assembly that is clamped in the welding fixture as shown in Figure 1.

The test piece assembly shall be cleaned in acetone prior to being clamped into the welding fixture. Copper foil strips, as shown in [Figure 1](#), shall be used to facilitate thermal transfer and prevent erosion of the fixture.

The welding procedure is specified in steps a) to f):

- a) Welding shall be initiated on the run on piece at a point sufficiently distant from the centre test piece such that a stable arc and a stable deposit shape are achieved before reaching the centre test piece.
- b) Welding shall be terminated when the trailing edge of the crater is on the run off piece but shall not exceed a distance of 25 mm from the centre test piece.
- c) After extinction of the arc, the clamp shall be released and the test piece assembly removed and immersed at $4\text{ s} \pm 1\text{ s}$ in an ice water bath. The test piece assembly shall be vigorously agitated or stirred in the ice water bath. After $20\text{ s} \pm 2\text{ s}$, the test piece assembly shall be transferred as quickly as possible and completely immersed in a low temperature bath containing, for example, methanol and solid carbon dioxide, denatured alcohol and solid carbon dioxide or liquid nitrogen. After removal of the specimen from the ice water, ice shall still be present in the bath.
- d) After a minimum of 2 min, the test assembly may be removed from the low temperature bath for cleaning and inspection. All slag and welding fume residue shall be removed by steel wire brushing. The run on and run off pieces shall be broken off from the centre test piece. The underside of this piece shall be examined to assess the uniformity and extent of heat tinting. Properly aligned and clamped test assemblies shall show parallel and uniform heat tinting of the underside of the centre test piece. Dark oxidation shall not extend to the edges of the underside of the centre test piece. If this entire operation is not completed within 60 s, the centre test piece shall be returned to the low temperature bath for a minimum of 2 min before completing these steps.
- e) Centre test pieces may be stored at -78 °C or lower in a methanol and solid carbon dioxide or denatured alcohol and solid carbon dioxide bath for a period of up to 72 h or at -196 °C in liquid nitrogen for a period of up to 21 days before analysis.
- f) For purposes of classifying welding consumables, during welding of the test assembly, the ambient absolute humidity shall be at least 3 g of water vapour per 1 000 g of dry air. (This corresponds to 20 °C and 20 % relative humidity.) When the relative humidity, measured using a sling hygrometer or other calibrated device, equals or exceeds this condition, the test shall be acceptable as demonstrating compliance with the requirements of this document provided the actual test results satisfy the diffusible hydrogen requirements of the applicable consumable classification standard. (The measurement of relative humidity can be easily converted to absolute humidity and reported as such.)

5.1.5 Recording of data

All relevant welding data, as shown on the data sheets, shall be recorded on the appropriate weld data sheet. Reference should be made to the suggested report forms for each welding process data sheet (see [Tables 2, 3, and 4](#)). Ambient conditions of temperature and humidity at the time of welding shall also be recorded and absolute humidity reported with the analytical results.

5.2 Welding procedures for the production of weld specimens

5.2.1 Summary

The operating parameters of the welding process under investigation shall be defined to produce a single weld bead on a test piece assembly as specified in [5.1](#). See [5.2.2](#) to [5.2.4](#) for specifications of the procedures for different welding processes.

5.2.2 Manual metal arc welding

5.2.2.1 Electrodes

The covered electrode to be tested shall be used in one of the ways a) or b).

- a) For purposes of classification, the electrode and the method of deposition of the weld shall be as specified in the standard with which the electrode complies.
- b) For purposes of investigation, the electrode and welding parameters shall be those given in the specific welding procedure. If no procedure has been given, then a current that is 90 % of the maximum suggested by the manufacturer shall be used.

When a pre-drying treatment is required, the time and temperature specified by the consumable manufacturer shall be used. If a range is given by the manufacturer, e.g. 300 °C to 350 °C, then the average shall be used and reported.

Electrodes with cracked or broken coatings shall not be used. Electrodes to be tested in the as received condition shall be taken from a freshly opened undamaged package. During any drying treatment, the electrodes shall not touch each other or the side of the oven. During any drying operation, a calibrated oven shall be used and the electrodes shall spend the full specified time at the drying temperature. Only electrodes under test shall be placed in the oven during this time. When the drying operation is complete, the electrode shall be cooled to ambient temperature in a container, e.g. a dried borosilicate glass tube sealed with a rubber bung. The electrode shall be used as soon as possible after it reaches ambient temperature, but not more than 1 h after removal from the oven unless securely sealed. Any electrodes removed from the drying oven and not then used shall not be re-dried and subsequently used for the test.

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When electrodes are to be tested in the as received condition from a hermetically sealed container, the electrodes shall be protected from moisture pickup once the seal is broken, until each can be welded. Some sealed containers are re-sealable. In such a case, each test electrode can be withdrawn individually and the container resealed while the withdrawn electrode is welded. If the container is not re-sealable, then all of the test electrodes shall be withdrawn when the seal is broken, and each electrode shall be individually placed in a dried borosilicate glass tube sealed with a rubber bung until the electrode is to be used for test.

5.2.2.2 Making the test welds

A copper fixture, such as that shown in [Figure 1](#), shall be used for the alignment and clamping of the test piece assembly. The fixture may incorporate water cooling channels in order to achieve a faster throughput of test pieces. Either test piece assembly A, assembly B, or assembly D may be used.

If the classification standard is silent on this matter, the following shall apply. The classification of covered electrodes is carried out using 4 mm diameter electrodes. In this case, the welding current shall be 15 A less than the maximum or 90 % of the maximum stated by the manufacturer, being maintained within a tolerance of ± 10 A. For an electrode with a diameter of 4 mm, the speed of welding shall be adjusted to produce an 8 g minimum weld deposit on the centre test piece of assembly A, a 3 g minimum weld deposit on the centre test piece of assembly B, or a 4 g minimum weld deposit on the centre test piece of assembly D, which is usually accomplished with an electrode consumption of between 1,2 cm and 1,3 cm per centimetre of weld. Record welding parameters and calculate heat input in accordance with ISO/TR 17671-1. For all consumable diameters other than the 4 mm specified above, the weld deposit sample mass shall be representative of good welding practice and appropriate for the diameter and process applied; no minimum weld deposit sample mass is specified.

Three or more test welds shall be made on three or more test piece assemblies using a new electrode for each weld. The deposit shall be made, without weaving, along the centre line of the test piece assembly, as shown in [Figure 1](#). The lengths of the run on and the run off pieces shall be 25 mm minimum. No burning off prior to testing shall be allowed. The run on deposit length shall not exceed 25 mm. The time spent in deposition shall be noted. Welding shall be terminated when the trailing edge of the crater is on the run off piece, but shall not exceed a distance of 25 mm from the centre test piece.

The method of using the welding fixture is specified in 5.1.4. When welding is completed, the weld specimen shall be quenched and stored as specified in 5.1.4, after which it shall be cleaned and analysed for hydrogen content as specified in 5.3.

At the time of welding, due to the influence of atmospheric moisture on the test results, for purposes of classifying covered electrodes, the arc length shall be maintained as short as possible consistent with maintaining a steady arc. For all purposes, the details listed in 5.2.2.3 shall be recorded.

5.2.2.3 Recording of welding data and results report form

The report sheet given in Table 2 provides full details of all the test variables that pertain to the test results.

5.2.3 Submerged arc welding

5.2.3.1 Electrode wire

The consumable solid or cored wire to be tested shall be used in one of the ways a) or b).

- a) For purposes of classification, the welding parameters shall be the same as those used in the preparation of the all weld metal test assembly for mechanical property determination using 4 mm electrode wire, with travel speed adjusted to provide a deposit mass on the centre test piece of 8 g minimum (assembly A), 3 g minimum for the smaller centre test piece (assembly C), or 4 g minimum for the smaller centre test piece (assembly D), unless otherwise specified in the classification document. Test piece assembly B is not acceptable for submerged arc welds. For all consumable diameters other than the 4 mm specified above, the weld deposit sample mass shall be representative of good welding practice and appropriate for the diameter and process applied; no minimum weld deposit sample mass is specified.
- b) For purposes of investigation, the electrode wire and welding parameters shall be those given in the specific welding procedure. The use of a solid wire that has been degassed in a vacuum or inert gas at 650 °C for 1 h facilitates the investigation of the effect of welding parameters, and type of flux and its drying procedure, upon the hydrogen content of the weld.

The arc energy for making the weld is restricted to a maximum of 3 kJ/mm.

5.2.3.2 Flux

When drying is required, the flux shall be dried in one of the ways a) or b).

- a) For purposes of classification, drying shall be done in accordance with the requirements of the standard with which the flux complies. Any preconditioning or drying of the flux shall be reported when an “H” designator is affixed to the flux classification.
- b) For purposes of investigation, drying shall be done in accordance with the appropriate recommendations.

Approximately 1 kg of flux is required for three welds. Drying shall be done in an open container placed in a calibrated drying oven set at the correct temperature. Flux depth should be limited in order to ensure that the entire mass of flux reaches the required temperature.

The flux shall spend the full specified time at the drying temperature and no other item(s) shall be placed in the oven during this time. When the drying treatment is complete, the flux shall be cooled to ambient temperature and used immediately. Alternatively, the flux shall be cooled in a sealed container and stored until required for use. Used flux shall not be recycled.