
**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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3690 was prepared by the International Institute of Welding, recognized as an international standardizing body in the field of welding in accordance with Council Resolution 42/1999.

This third edition cancels and replaces the second edition (ISO 3690:2000), which has been technically revised.

Requests for official interpretations of any aspect of this International Standard should be directed to the ISO Central Secretariat, who will forward them to the IIW Secretariat for an official response.

This corrected version of ISO 3690:2012 incorporates the following corrections:

- a) to comply with ISO quality documentation, references to Commission II and to TC 44/SC 3 have been removed from paragraph 5 of this foreword;
- b) the quality of Figures 1 and 3 has been improved in terms of resolution and presentation.

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

1 Scope

This International Standard 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 metal.

The techniques specified in this International Standard 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.

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2 Normative references (standards.iteh.ai)

The following referenced documents are indispensable for the application 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, *Quantities and units — Part 1: General*

3 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.

4 Test procedures

4.1 Production of weld specimens

4.1.1 Summary

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

4.1.2 Welding fixture

An example of a suitable welding fixture to provide uniform test pieces for the welding processes specified in 4.2 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 4.1.4 c). The surface temperature of the fixture shall be between ambient and $25\text{ }^{\circ}\text{C}$ 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.

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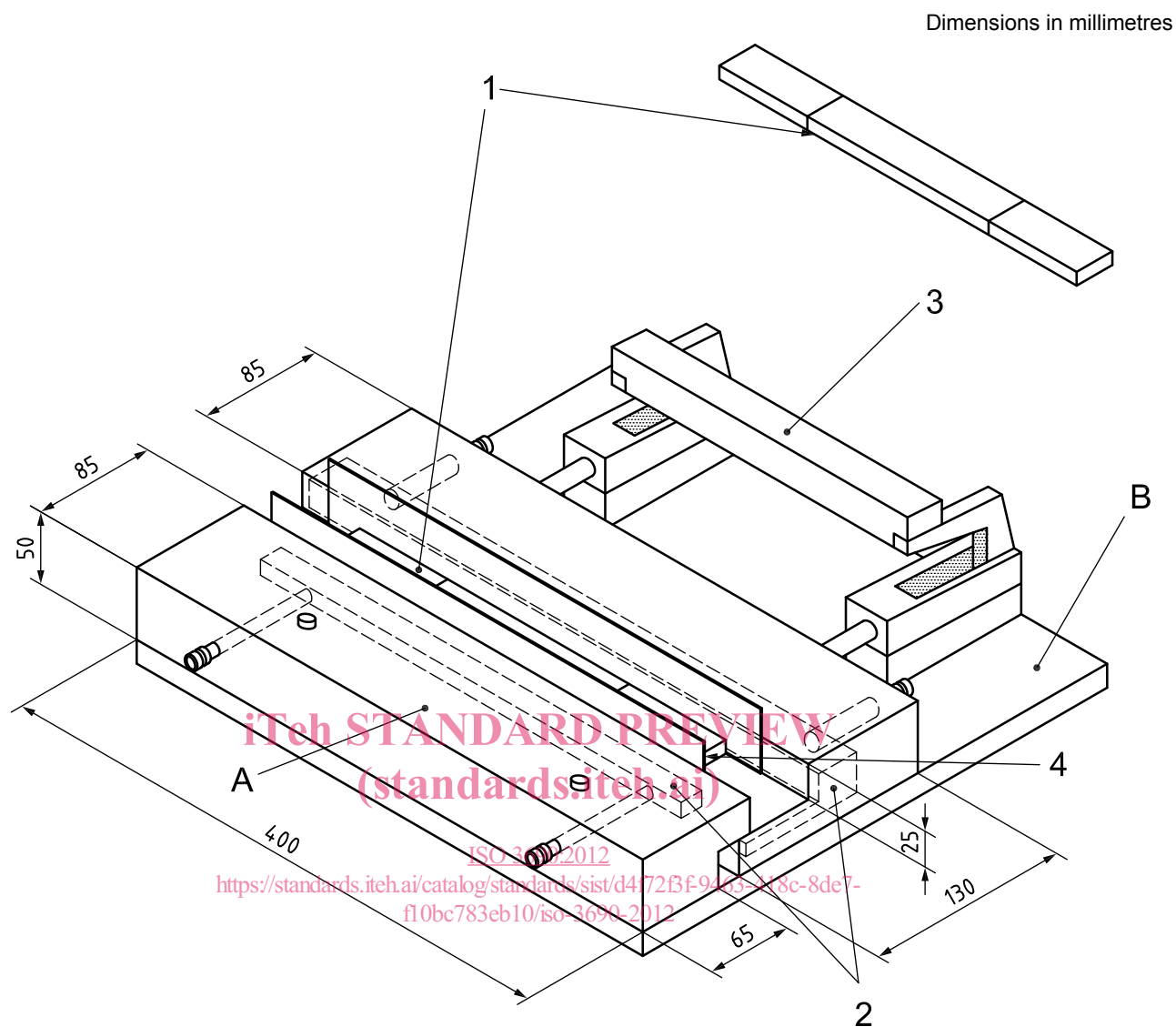


Figure 1 — Example of a suitable welding fixture and test piece assembly for weld deposits

4.1.3 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 for assembly A, assembly B, or assembly C, 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 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 4.1.4 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 $650\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ 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 or assembly C.



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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

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
$l_a \geq 25$ mm and $l_b \geq 25$ mm: manual metal arc welding				
$l_a \geq 50$ mm and $l_b \geq 50$ mm: other welding processes				
^a Comparable to the specimen geometry according to AWS A4.3-93[5].				

Figure 2 — Dimensions of the weld test assembly

4.1.4 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 4.2, 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).

- c) 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.
- d) 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.
- e) 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. After $20\text{ s} \pm 2\text{ s}$, the test piece assembly shall be transferred 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.
- f) 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.
- g) 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.
- h) 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 International Standard 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.)

4.1.5 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 1, 2, and 3). Ambient conditions of temperature and humidity at the time of welding shall also be recorded and absolute humidity reported with the analytical results.

4.2 Welding procedures for the production of weld specimens

4.2.1 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 4.1. See 4.2.2 to 4.2.4 for specifications of the procedures for different welding processes.

4.2.2 Manual metal arc welding

4.2.2.1 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 predrying 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 redried and subsequently used for the test.

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 resealable. 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 resealable, 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.

4.2.2.2 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 or assembly B 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 assembly A or a 3 g minimum weld deposit on the centre test assembly B, 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 of 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 4.1.4. When welding is completed, the weld specimen shall be quenched and stored as specified in 4.1.4, after which it shall be cleaned and analysed for hydrogen content as specified in 4.3.

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 4.2.2.3 shall be recorded.

4.2.2.3 Recording of welding data and results report form

The report sheet given in Table 1 provides full details of all the test variables that pertain to the test results.

Table 1 — Report form (diffusible hydrogen, manual metal arc)

Investigating laboratory:		Date:	
Investigator's name:			
Brand name & electrode maker:		Batch No.:	
Type of electrode:	Electrode designation:		
Diameter of electrode (mm):	Overall length of electrode (mm):		
Drying treatment: °C for h			
Electrode polarity (d.c. + ve, d.c. – ve or a.c.):			
Relative humidity % and temperature °C at the welding station during welding			
Hydrogen extraction temperature: °C			
Hydrogen extraction time: days h min			
Type of test piece assembly (A or B):			
Number of test piece:	1	2	3
Voltage, V; a.c. or d.c.:			
Current, A:			
Welding time, s:			
Weld length, mm:			
Heat input, kJ/mm:			
Electrode length used, mm:			
Run-on length, mm:			
Mass of deposited metal on test piece, g:			
Test piece to crater distance, mm:			
Diffusible hydrogen			
	1	2	3
a) H_D , ml/100 g of deposited metal:			Average
b) H_F , ppm of fused metal:			
Other test details not included above:			