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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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Contents

Foreword

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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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This document was prepared by IIW, International Institute of Welding, Commission II.

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

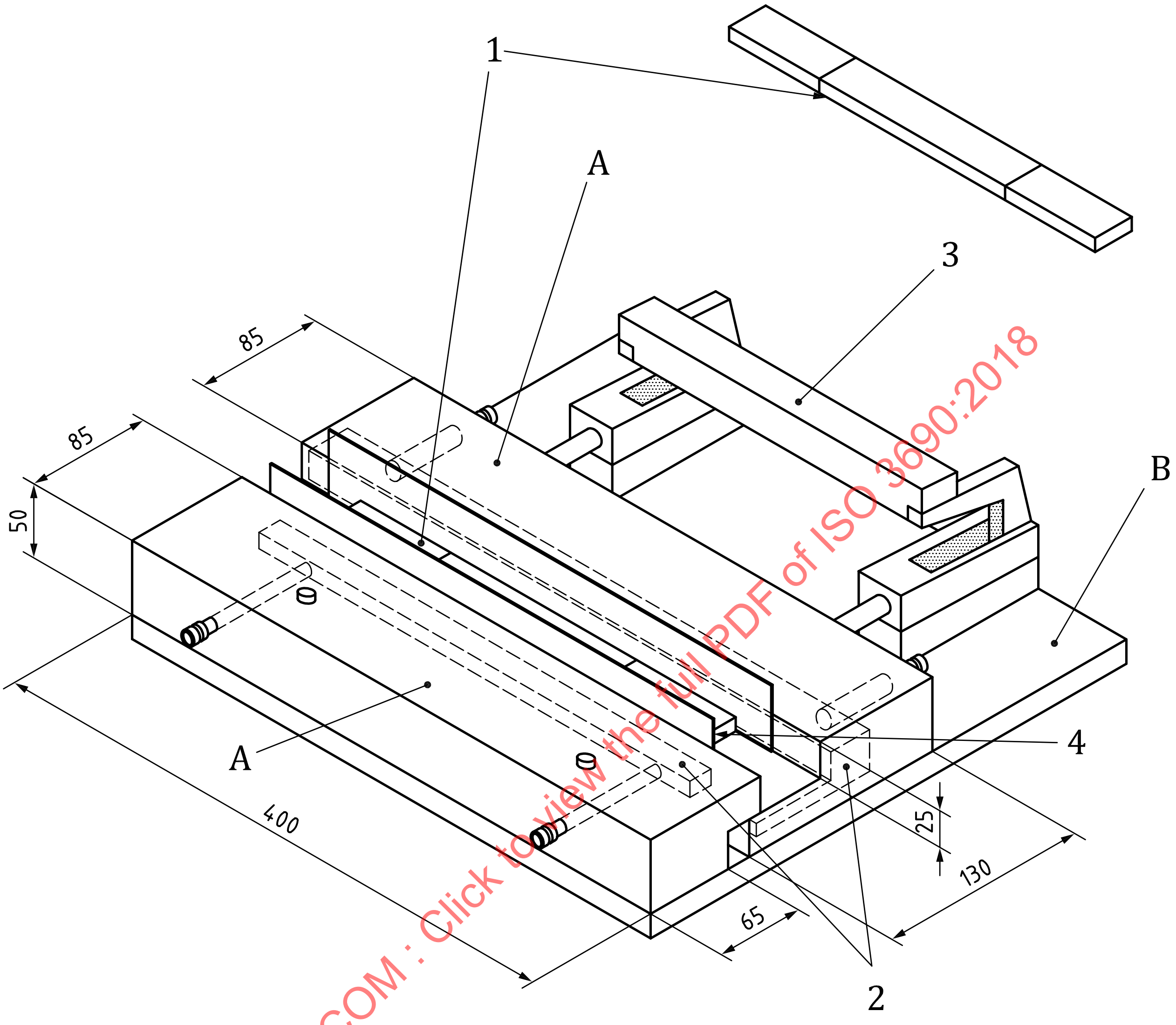
The welding consumable to be tested is used to deposit a single weld bead, which is rapidly quenched and subsequently stored at −78 °C or lower until analysis. Cleaning and slag removal are performed on the chilled specimen.

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.

Dimensions in millimetres



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

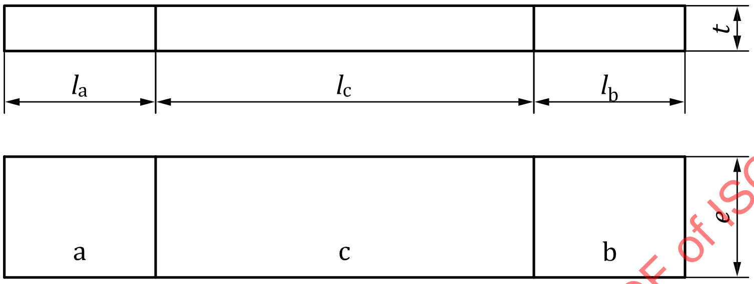
Figure 1 — Example of a suitable welding fixture and test piece assembly for weld deposits

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 ±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 °C ± 10 °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

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

$l_a \geq 25$ 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] 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 s ± 1 s in an ice water bath. The test piece assembly shall be vigorously agitated or stirred in the ice water bath. After 20 s ± 2 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.

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.

Table 2 — 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 or D):				
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	Average
a) H_D, ml/100 g of deposited metal:				
b) H_F, ppm of fused metal:				
Other test details not included above:				

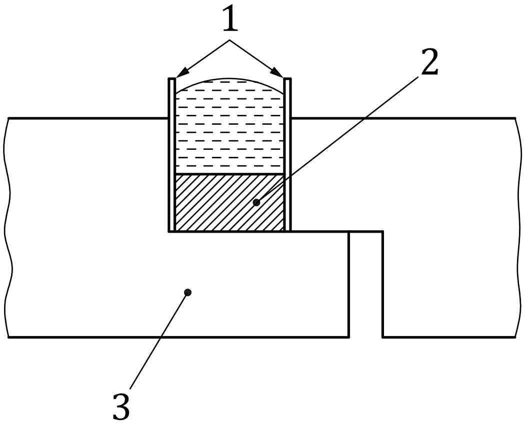
5.2.3.3 Making the test welds

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 C, or assembly D may be used.

The centre piece remains the same size as specified in 5.1.3, but is aligned with longer run on and run off pieces of 50 mm minimum. The preparation, degassing, and use of the test piece assembly are specified in 5.1.3. The flux is kept at a predetermined constant depth of 25 mm or as recommended by the manufacturer. One method of controlling flux depth is by levelling off along the top of the copper foil inserts shown in Figure 3. The flux depth of 25 mm or a different flux depth specified by the flux manufacturer, determines whether the dimension of the copper foil should be modified in order to achieve the specified flux depth. At the end of the copper foil, there shall be a suitable piece of copper foil to contain the flux.

Three or more test welds shall be made on different test piece assemblies. The deposit shall be along the centre line of the test piece assembly. The time spent in deposition shall be noted. The trailing end of the crater shall be entirely on the run off piece, but no further than 25 mm from the centre test piece for submerged arc welding. No length for the run on portion of the weld deposit is specified, but the length shall be sufficient to achieve arc and deposit stability before reaching the centre test piece.

After extinction of the arc, and without any delay, the test piece assembly shall be released from the fixture and the test piece quenched, cleaned, and stored as specified in 5.1.4.



Key

1 1 mm copper foil, of dimensions 35 mm × 300 mm or 37 mm × 300 mm (for flux depth of 25 mm)
2 test piece assembly
3 welding fixture

Figure 3 — Example of the use of copper foil to maintain constant flux depth

5.2.3.4 Recording of welding data and results report form

The report sheet given in Table 3 provides full details of all the test variables that pertain to the test results.

Table 3 — Report form (diffusible hydrogen, submerged arc)

Investigating laboratory:	Date:		
Investigator's name:			
Electrode diameter, mm:	Electrode designation:		
	Flux designation:		
Brand name & electrode maker:	Batch No.:		
Brand name & flux maker:	Batch No.:		
Flux drying temperature and time: ____ °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 C or D):			
Number of test piece:	1	2	3
Voltage, V; a.c. or d.c.:			
Current, A:			
Welding time, s:			
Weld length, mm:			
Welding speed, mm/s:			
Heat input, kJ/mm:			
Wire feed speed, mm/s:			
Contact tube distance, mm:			

Table 3 *(continued)*

Run-on length, mm:				
Mass of deposited metal on test piece, g:				
Test piece to crater distance, mm:				
Diffusible hydrogen				
	1	2	3	Average
a) H_D, ml/100 g of deposited metal:				
b) H_F, ppm of fused metal:				
Other test details not included above:				

5.2.4 Tubular cored electrode with or without gas shield and wire electrode with gas shield

5.2.4.1 Filler material

The filler material 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 a 1,2 mm wire electrode, with travel speed adjusted to obtain a weld deposit mass on the centre test piece of 8 g minimum (assembly A), 3 g minimum for the smaller centre test piece (assembly B), or 4 g minimum for the smaller centre test piece (assembly D), unless otherwise specified in the classification document. It is well established that diffusible hydrogen results from tubular cored electrodes are strongly affected by the electrode extension. Care shall be taken that the contact tip to work distance used for the diffusible hydrogen test is the same as that used in preparing the all weld metal test coupon for mechanical property determination. For all consumable diameters other than the 1,2 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 investigation purposes, the filler material and welding parameters shall be those given in the appropriate welding procedure. If a current range is given by the manufacturer, then the average shall be used.

5.2.4.2 Shielding gas

The shielding gas shall be of a welding grade as specified in ISO 14175. The shielding gas used and gas flow shall be in accordance with the appropriate recommendations. Details of the shielding gas composition and flow shall be recorded on the report form. For investigation purposes, it may sometimes be necessary to dry the shielding gas in order to remove moisture. If this is done, then the moisture content of the gas shall be measured and reported.

5.2.4.3 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.

The run-on and run-off pieces of the test piece assemblies shall have a minimum length of 50 mm. The test piece assembly shall be clamped in the fixture using annealed copper foil as shown in Figure 1.

Three or more test welds shall be made on different test piece assemblies. The deposit shall be along the centre line of the test piece assembly and weld deposit masses shall be as specified in 5.2.4.1. The time spent in deposition shall be noted. The trailing end of the crater shall be on the run off piece, but at a maximum distance of 25 mm from the centre test piece. No length for the run on portion of the weld deposit is specified, but the length shall be sufficient to achieve arc and deposit stability before reaching the centre test piece.

After extinction of the arc and without any delay, the test piece assembly shall be released from the fixture and the test piece quenched, cleaned, and stored as specified in 5.1.4.

5.2.4.4 Recording of welding data and results report form

The report sheet given in Table 4 provides full details of all the test variables that pertain to the test results.

Table 4 — Report form (diffusible hydrogen, MIG, MAG, TIG or cored electrode)

Investigating laboratory:		Date:
Investigator's name:		
Type of filler material:	Drying treatment: ____ °C for ____ h	
Filler material designation:		Gas cup i.d., mm:
Brand name and electrode maker:	Batch No.:	Diameter of filler material, mm:
Shielding gas:		
Shielding gas flow, l/min:		
Electrode polarity (d.c. +ve, d.c. −ve or a.c.):		Details of tungsten electrode, if any :
		Make:
Relative humidity ____ % and temperature ____°C at the welding station during welding		Diameter, mm:
Hydrogen extraction temperature: ____ °C		Cone angle:
Hydrogen extraction time: ____ days ____ h ____ min		Designation
Type of test piece assembly (A or B or D):		

Number of test piece:	1	2	3
Voltage, V; a.c. or d.c.:			
Current, A:			
Welding time, s:			
Weld length, mm:			
Welding speed, mm/s:			
Heat input, kJ/mm:			
Wire feed speed, mm/s:			
Contact tube distance, mm:			
Run-on length, mm:			
Mass of deposited metal on test piece, g:			
Test piece to crater distance, mm:			

Diffusible hydrogen				
	1	2	3	Average
a) H_D, ml/100 g of deposited metal:				
b) H_F, ppm of fused metal:				
Other test details not included above:				

5.3 Measurement of hydrogen in the test weld

5.3.1 General

The measurement method shall ensure that only diffusible hydrogen is released from the test specimen and is collected, and material dependent activation of potential trapped hydrogen sources shall be avoided. Two methods of diffusible hydrogen analysis are specified in this document, displacement of mercury and thermal conductivity detection.

NOTE Recommendations and restrictions in regard to older methods of measurement using mercury are given in Annex A.

5.3.2 Displacement of mercury method

The gas collection apparatus specified in this clause is known as a Y tube and mercury shall be used as the collecting fluid. Other designs of collecting may be used, provided the same principles as for the Y tube are used. The gas collection apparatus specified is for use with test centre piece assembly B or assembly C (see Figure 2).

5.3.2.1 Preparation of gas burette

The volume of mercury required to fill the size of a gas burette illustrated in Figure 4 is about 110 ml. The mercury shall be free of contaminants. The mercury shall be poured into the wide limb of the gas burette, the two way vacuum stopcock shall be fitted and vacuum applied. Air shall be removed by laying the gas burette on a flat surface so as to allow an unrestricted application of the vacuum to the top of the capillary tube. The gas burette shall be slowly raised to the vertical position and air admitted to the wide limb by rotating the stopcock. It is important that there is no air bubble at the top of the capillary tube. If air is present, then the evacuation process shall be repeated until a final check confirms the absence of air. The vacuum stopcock shall be removed from the wide limb of the gas burette.

5.3.2.2 Test piece cleaning and handling

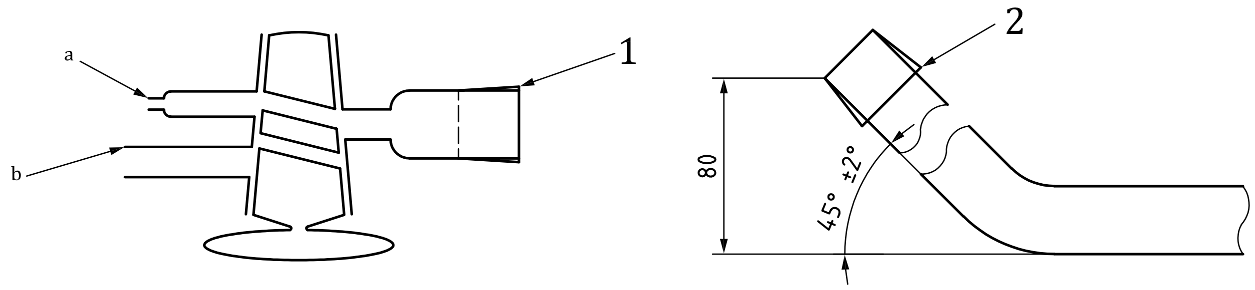
The centre test piece shall be cleaned by thorough brushing to remove all slag and oxide using a steel wire brush, in good condition, with intermittent periods of cooling (2 min minimum). Each interval spent outside the cooling bath during this operation shall not exceed 60 s [see also 5.1.4 d) and 5.1.4 e)].

5.3.2.3 Test piece loading

The following part of the procedure shall be carried out as quickly as possible, taking not more than approximately 2 min.

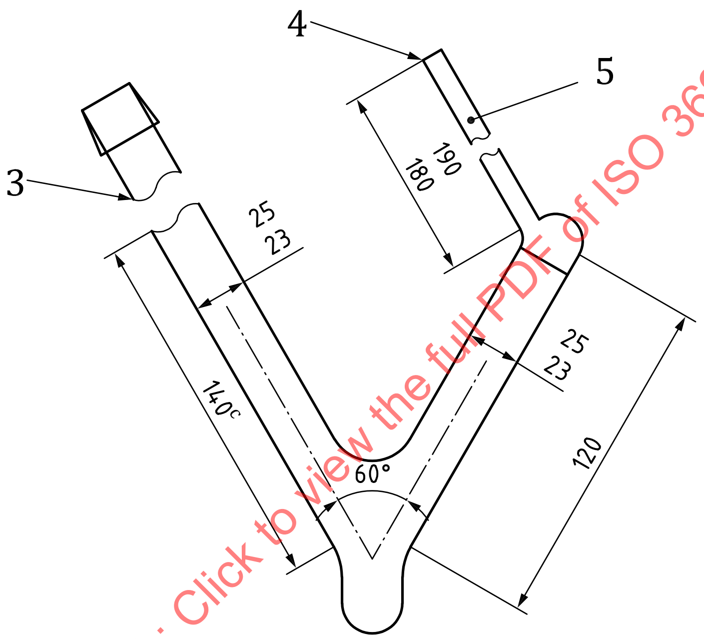
The test piece shall be removed from the storage coolant and raised to slightly above 0 °C. This can be conveniently achieved by immersing the test piece in water until the ice which forms initially has detached or melted.

Dimensions in millimetres



a) Two way glass vacuum stopcock

b) Side view of bent arm 3 in c)



c) Overall view of Y tube

Key

1	29/32 socket
2	29/32 cone
3	arm bent up 45° [see Figure 4 b)]
4	seal (flat inside)
5	precision bore tube
a	To air.
b	To vacuum.
c	Straight portion before bend.

NOTE Not to scale.

Figure 4 — Diffusible hydrogen collecting apparatus, Y tube

Following a rinse with acetone and drying in a jet of air, the test piece shall be transferred to the wide limb of the burette. The two way vacuum stopcock shall be closed and the burette evacuated. Acetone and traces of condensed water evaporate from the surface of the test piece and are removed with the evacuated air. Using a magnet, the test piece shall be manoeuvred into position under the capillary tube by carefully lowering the burette towards the horizontal position until it just floats along the mercury surface. It is essential that care be taken to prevent air from entering the capillary tube when the burette is raised to the vertical position and air is admitted to the wide limb. The two way stopcock shall be removed and the wide limb closed by means of a stopper or a glass cap, for example, to prevent the release of traces of mercury vapour.

Diffusible hydrogen is evolved from the test piece and collects in the capillary tube.

WARNING — Metallic mercury and mercury vapours are hazardous and can be absorbed into the body by inhalation, ingestion, or contact with the skin. All precautions involving the handling of mercury should be observed, which include but are not limited to the following:

a) the diffusible hydrogen test apparatus should be located under a fume hood, and any steps involving the handling of mercury should be performed under a fume hood;

b) the apparatus should be located on a tray or counter with a raised lip to contain any possible spills;

c) plastic or rubber gloves should be used at all times when handling mercury or mercury contaminated samples and equipment;

d) any mercury spill should be cleaned up immediately.

5.3.2.4 Analytical procedure

The test piece shall be maintained at 25 °C ± 5 °C, until there is no increase in calculated hydrogen volume, corrected to standard temperature and pressure (STP), i.e. 0 °C and 760 mmHg[1]), on successive days. “No increase” can be understood as allowing for a change, over 24 h, of no more than 1 % of the total volume collected. The length of the hydrogen gas column and the head of mercury shall be measured using a cathetometer. Room temperature and atmospheric pressure shall be measured and recorded. The volume of hydrogen collected, corrected to STP, V, in millilitres, is given by Formula (1):

$$V = \frac{273 \times (p - h) \pi r^2 C}{760 \times (273 + T) \times 1\,000} \quad (1)$$

where

C is the length of the gas column above the mercury, in millimetres;

p is the atmospheric pressure, in millimetres of mercury;

h is the differential head of mercury between the two limbs of the Y tube, in millimetres;

r is the inside radius of the capillary tube, in millimetres;

T is the room temperature at the time of hydrogen measurement, in degrees Celsius.

When evolution has ceased, the test piece shall be removed from the apparatus and its final mass, m_2, in grams, shall be determined, to the nearest 0,01 g. All the relevant data shall be recorded.

5.3.3 Thermal conductivity detector method

5.3.3.1 General

In general, systems utilizing a thermal conductivity detector fall into one of two types: the first is hot carrier gas extraction where the specimen is heated at a relatively high temperature (up to 400 °C) and the diffusible hydrogen is evolved from the specimen and measured continuously. The second type is where the specimen is loaded into a suitable collection chamber that is heated at a relatively lower temperature (typically between 45 °C and 150 °C). Quantification of the hydrogen collected is performed as a separate step, typically utilizing a gas chromatography device.

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1) 760 mmHg ≡ 101,325 kPa.

It is not the objective of this document to specify the several systems that are based on the thermal conductivity detector (TCD), e.g. gas chromatography or hot carrier gas extraction, that are available for the measurement of hydrogen in metals. However, it is important to note that any alternative system incorporating the facility for measuring the hydrogen in weld metal shall provide a proper correlation, in terms of accuracy and reproducibility. Particularly, the calibration procedures may be different for the individual measurement systems typically utilizing gas chromatography and a TCD. The manufacturer's instructions shall be followed for the gas extraction and thermal conductivity detection equipment. This device may be a gas chromatography device. This method is valid for the centre test piece assembly A, B, C, or D.

5.3.3.2 Test piece cleaning and handling

The centre test piece shall be cleaned by thorough brushing to remove all slag and oxide using a steel wire brush, in good condition, with intermittent periods of cooling. The intervals spent outside the cooling bath during this operation shall not exceed 60 s [see also 5.1.4 d) and 5.1.4 e)].

5.3.3.3 Test piece loading

The centre test piece shall be removed from the methanol and solid carbon dioxide bath, denatured alcohol and solid carbon dioxide bath or liquid nitrogen bath and raised to slightly above 0 °C. This can be conveniently achieved by immersing the test piece in water until the ice which forms initially has detached or melted. The test piece shall be rinsed in acetone, dried in an air jet, and loaded into a suitable container for collection of hydrogen. The container shall be purged and filled with an inert gas such as argon and isolated from the atmosphere.

5.3.3.4 Measurement method by hot carrier gas extraction

Hydrogen extraction is accomplished in this procedure within a short period of time (rapid method). The manufacturer's instructions shall be followed to operate the equipment. After warming the specimen in accordance with 5.3.3.3, it shall be loaded into a suitable container for the collection of hydrogen. The test piece and container may be heated up to a maximum of 400 °C for measurement of the diffusible hydrogen content. Collection and measurement of the hydrogen occurs continuously until all of the diffusible hydrogen is quantified. (See footnote to Table 5.) Exceeding the degassing temperature of 400 °C can lead to an activation of further hydrogen traps, thus elevating the apparent diffusible hydrogen results.

5.3.3.5 Measurement method for collection and subsequent measurement of hydrogen

After warming the specimen per 5.3.3.3, it shall be loaded into a suitable container for the collection of hydrogen. The container shall be purged and filled with an inert gas such as argon and sealed to the atmosphere. The container shall be transferred to an oven or other suitable heating device for collection of hydrogen per the operating conditions specified in Table 5.

Upon completion of the heating and collection period, the container shall be cooled to room temperature and analysed for hydrogen. This can be accomplished by analysing either the entire quantity of hydrogen evolved from the test piece or an aliquot that can be apportioned to the entire quantity. A TCD shall be used to quantify the amount of hydrogen present in the outgas mixture.

Conversion of the measured value to give the hydrogen volume at STP, V_H, in millilitres, shall be made using Formula (2):

$$V_H = \frac{273}{(273+T)} \times \frac{pV}{760} \tag{2}$$

where

p is the pressure of the measured hydrogen volume, in millimetres of mercury;

V is the measured volume of hydrogen, in millilitres;

T is the temperature of the measured hydrogen volume, in degrees Celsius.

The test piece shall be removed from the container, weighed, and the mass, m_2, recorded to the nearest 0,1 g for assembly A centre pieces or to the nearest 0,01 g for assembly B, C, or D centre test pieces.

Table 5 — Minimum holding time at a given temperature for complete hydrogen evolution

Minimum extraction time h	Specimen temperature[a] °C
0,35	400 ± 3
0,4	390 ± 3
0,5	360 ± 3
1	285 ± 3
2	225 ± 3
3	195 ± 3
4	175 ± 3
5	160 ± 3
6	150 ± 3
8	140 ± 3
10	125 ± 3
12	120 ± 3
14	115 ± 3
15	110 ± 3
18	100 ± 3
36	70 ± 3
64	50 ± 3
72	45 ± 3

[a] The temperatures shown above are the temperature of the specimen, not necessarily that indicated by the equipment. Sufficient calibration tests shall be conducted to determine the time necessary to reach the desired temperature and that the time at temperature is sufficient to release substantially all of the diffusible hydrogen (see 5.3.2.4).

5.3.4 Calibration

The calibration shall be carried out by supplying a known hydrogen volume to the carrier gas hot extraction system over the working area of interest. The analysis instruments should preferably incorporate different closed gas calibration units with different volumes. The apparatus shall be assembled and operated following the instructions of the manufacturer. Depending on the analysis instrument, a sufficiently long gas flow time shall be assured for stabilization prior to analysis. During normal instrument use, any existing calibration shall be checked by gas dosing before analysis is attempted. The instrument preparation procedure shall also be adhered to. Checks shall be made at the start and completion of a series of analyses in order to ensure that no drift has occurred in the instrument calibration. The interval between drift checks shall not exceed 4 h. The detector shall be calibrated over the appropriate range of hydrogen. In addition, for the hot extraction method sufficient calibration tests shall be conducted to determine the time necessary to reach the desired temperature shown in Table 5 and that the time at temperature is sufficient to release substantially all of the diffusible hydrogen (see 5.3.3.4).

5.3.5 Linearity

The linearity of response of the instrument may be judged by using linear regression analysis to fit the calibration data and then calculating the correlation, R^2. Values of R^2 close to unity indicate a high degree of correlation.

The range of hydrogen contents to be measured ranges from 0,05 ml to significantly over 1 ml at STP, when the entire diffused volume of hydrogen gas is captured and analysed. The range of hydrogen contents to be measured is significantly less when analysis is based on methods utilizing a gas sample taken from the collection container. Hydrogen injection shall cover the applicable range in order to confirm linearity of response.

5.3.6 Calculation and expression of results

5.3.6.1 Diffusible hydrogen in deposited metal, H_D

The volume at STP of diffusible hydrogen in deposited metal, H_D, in millilitres per 100 g, shall be calculated from Formula (3):

$$H_D = V_{STP} \times \frac{100}{m_2 - m_1} \tag{3}$$

where

V_{STP} is the volume of hydrogen gas at STP, in millilitres,

m_2 is the final mass of the test piece with deposited metal, in grams;

m_1 is the initial mass of the test piece, in grams.

5.3.6.2 Diffusible hydrogen in fused weld metal, H_F

In order to assess the diffusible hydrogen content in a welded joint, it is necessary to determine the diffusible hydrogen in the fused weld metal. In the calculation of the diffusible hydrogen in fused weld metal, not only the deposited metal is considered, but also the fused portions of the base material.

Thus, in determining the fused weld metal, the respective total weld volume, and hence also the hydrogen pickup in the base material, is taken into account. In general, for a given test sample, H_F is of the order of 50 % of H_D.

If the hydrogen content is required in terms of concentration in the fused metal, it is necessary to measure the cross-sectional area of the fused metal and of the deposited metal. These shall be measured on the ends of the test piece by using an enlarged tracing or photograph, or an image-analysing microscope, and then averaging the results. Diffusible hydrogen in the fused weld, H_F, in parts per million by mass [ppm], shall be calculated from Formula (4):

$$H_F = H_D \times 0{,}9 \times \frac{A_D}{A_F} \tag{4}$$

where

H_D is the diffusible hydrogen concentration in the deposited weld metal, in millilitres per 100 g;

A_D is the average area of deposited weld metal [see Figure 5 a)];

A_F is the average area of fused metal [see Figure 5 b)];

0,9 is the conversion factor to change ml/100 g to ppm.

a) A_D - average area of deposited weld metal **b) A_F - average area of fused metal**

Figure 5 — Deposited weld metal and fused metal

5.3.7 Reporting of results

All data that are relevant to the interpretation of results shall be reported on the analysis data sheet given in Table 6 as appropriate for the method. The hydrogen concentration of each individual weld sample shall be calculated to one or two significant figures (as appropriate). For the purposes of this document, the average value of the hydrogen concentration of triplicate welds, at a minimum, shall be calculated to the same number of significant figures and then rounded and reported to the nearest whole number for reporting in millilitres per 100 g.

The report forms given in Tables 2, 3, and 4 of this document are used to report details of the welding consumable, the welding parameters, and test conditions for each set of triplicate welds. The results of the hydrogen measurements shall be recorded on the same forms.

Where applicable, rounding shall be in accordance with 5.5.

5.4 Measurement of total hydrogen content in the weld metal — Rapid methods

Specification of an elevated degassing temperature for the total hydrogen content (diffusible plus trapped) is only possible with knowledge of the activation energy of the hydrogen traps depending on the material to be investigated. For determination of the total hydrogen content, a melt extraction method can be applied.

Higher than 400 °C, analysis for total hydrogen, which includes residual hydrogen, can be achieved. The condition of the sample surface has a marked effect upon the measured hydrogen volume when methods involving heating above about 500 °C are used.

5.5 Rounding procedure

Actual test values obtained shall be subject to ISO 80000-1:2009, B.3, Rule A. If the measured values are obtained by equipment calibrated in units other than those of this document, the measured values shall be converted to the units of this document before rounding. If an average value is to be compared to the requirements of this document, rounding shall be done only after calculating the average.