

Diffusible hydrogen —

Part 1: Method for determination of hydrogen in manual metal-arc weld metal using 3 day collection

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Welding Standards Committee (WEE/-) to Technical Committee WEE/39, upon which the following bodies were represented:

British Association for Brazing and Soldering
 British Railways Board
 British Shipbuilders
 British Steel Industry
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Foreword

This British Standard has been prepared under the direction of the Welding Standards Committee and is being published in separate Parts covering different methods of determining diffusible hydrogen.

This Part of BS 6693 was first published as Appendix C of BS 639:1976 and was subsequently called up by other British Standards, for example BS 5135.

Although this method is becoming obsolete it has been retained for the present as it is the basis of useful and accepted data in BS 5135 and its withdrawal in favour of a method involving collection of hydrogen until no further gas was evolved (BS 6693-2) could cause some initial confusion.

This Part of BS 6693 is closely related to ISO 3690 published by the International Organization for Standardization (ISO).

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 6693 describes a method for the determination of hydrogen in manual metal-arc weld metal which is evolved in a three day collection period.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

The electrode to be tested is used to deposit a single weld bead which is rapidly quenched. Both the welding and quenching procedures are carefully controlled. The specimen so produced is maintained at room temperature for a sufficient time to release its content of diffusible hydrogen, which is collected over mercury and measured by a volumetric method.

3 Materials and apparatus

3.1 Parent plate material. The test piece assembly shall be prepared from a semi-killed grade of steel containing not more than 0.20 % C and not more than 0.05 % S. Prior to use the test piece shall be degassed at 650 °C for 1 h.

3.2 Electrodes. The electrode to be tested shall be of 4 mm core wire diameter, or of 3.15 mm diameter in the case of an electrode having a nominal efficiency higher than 130 %. The electrode shall be dried at 250 °C for 2 h.

3.3 Welding fixture. A copper jig, as shown in Figure 1, shall be used for the alignment and clamping of the test piece assembly.

3.4 Apparatus for determination of diffusible hydrogen. An example of a gas burette for the measurement of cold extracted gas is shown in Figure 2. Burettes of other designs may be employed, provided that the following requirements are fulfilled.

- a) Mercury shall be used as the confining liquid.
- b) It shall be possible to maintain the sample under vacuum for a brief period, as specified under 4.3 d), to remove any trace of foreign gases trapped on the fractured surfaces of the sample.

NOTE In burettes consisting of a single limb, this may be achieved through manipulation of the mercury level and the stopcock, any gas released during the brief period of surface degassing being swept out of the burette before the measurements.

- c) The volume of collected gas shall be measured to an accuracy of at least 0.05 mL (s.t.p.).

4 Procedure

4.1 Test piece assembly

Duplicate sets of test pieces having a cross section of 10 mm × 15 mm shall be used for each type of electrode to be tested. A bead of 100 mm overall length shall be deposited along the centreline of each test piece assembly. No burning-off prior to testing is allowed.

The test piece assembly shown in Figure 1 consists of run-on and run-off pieces, and a central sample section of 30 mm total length. Duplicate determinations shall be made using the entire length of this centre section. It may be divided into four specimens of 7.5 mm length each as indicated in Figure 1, or two specimens of 15 mm length each, or one specimen of 15 mm and two specimens of 7.5 mm length. The selection shall be made from the following three optional combinations.

- a) Nos. 1 and 4 (2 × 7.5 mm) analysed jointly. Nos. 2 and 3 (2 × 7.5 mm) analysed jointly.
- b) Nos. 1 and 4 (2 × 7.5 mm) analysed jointly. Central specimen (15 mm) analysed separately.
- c) Two specimens (15 mm each) analysed separately.

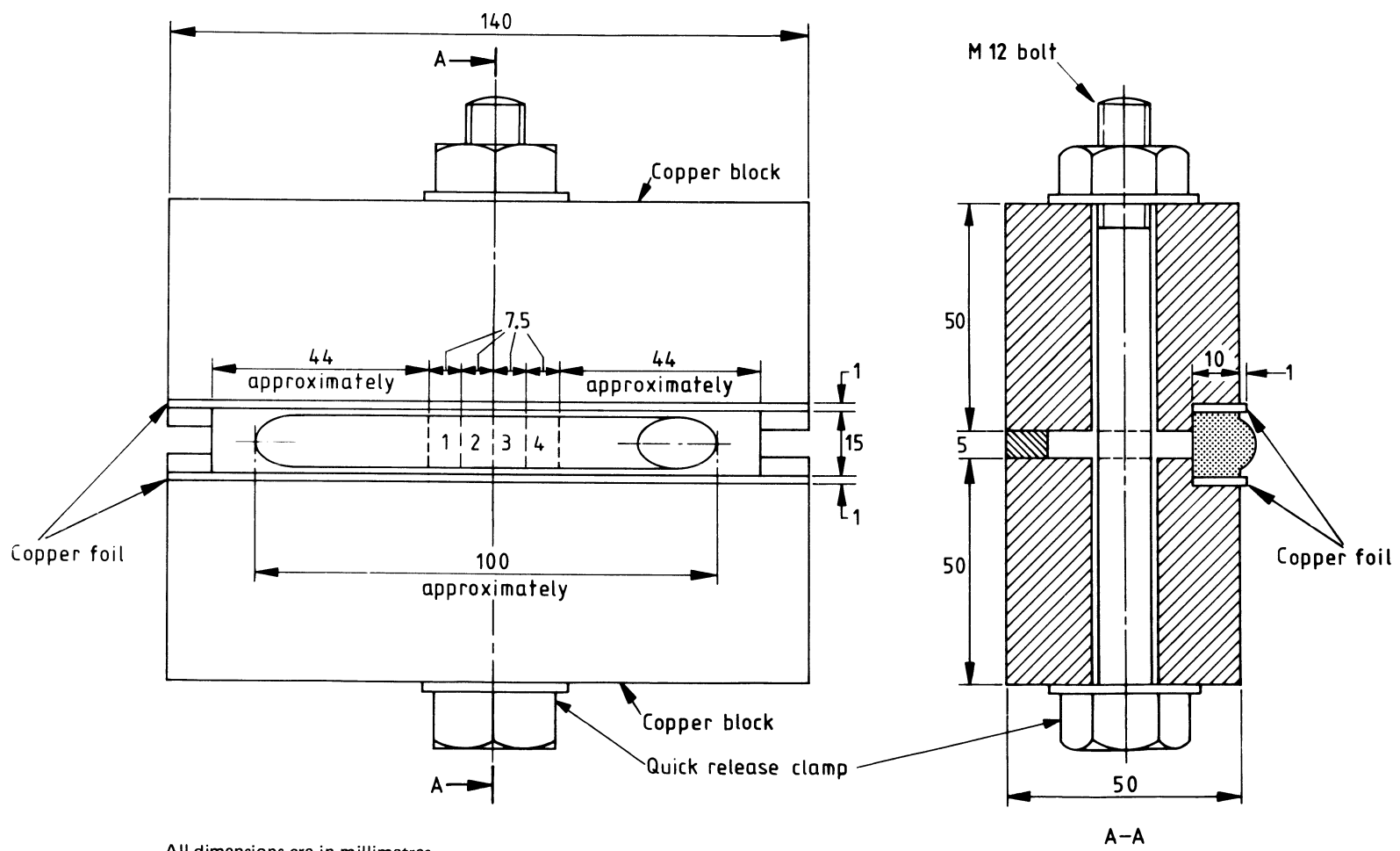
The test piece dimensions shown in Figure 1 shall be maintained within the limits of ± 0.25 mm. Each set, comprising run-on and run-off pieces and the central section, shall be finished in one operation of grinding so as to ensure a uniform width. The faces of the cross sections shall be machined to ensure good contact between adjacent pieces.

The central specimens shall be marked and weighed to the nearest 10 mg.

4.2 Welding

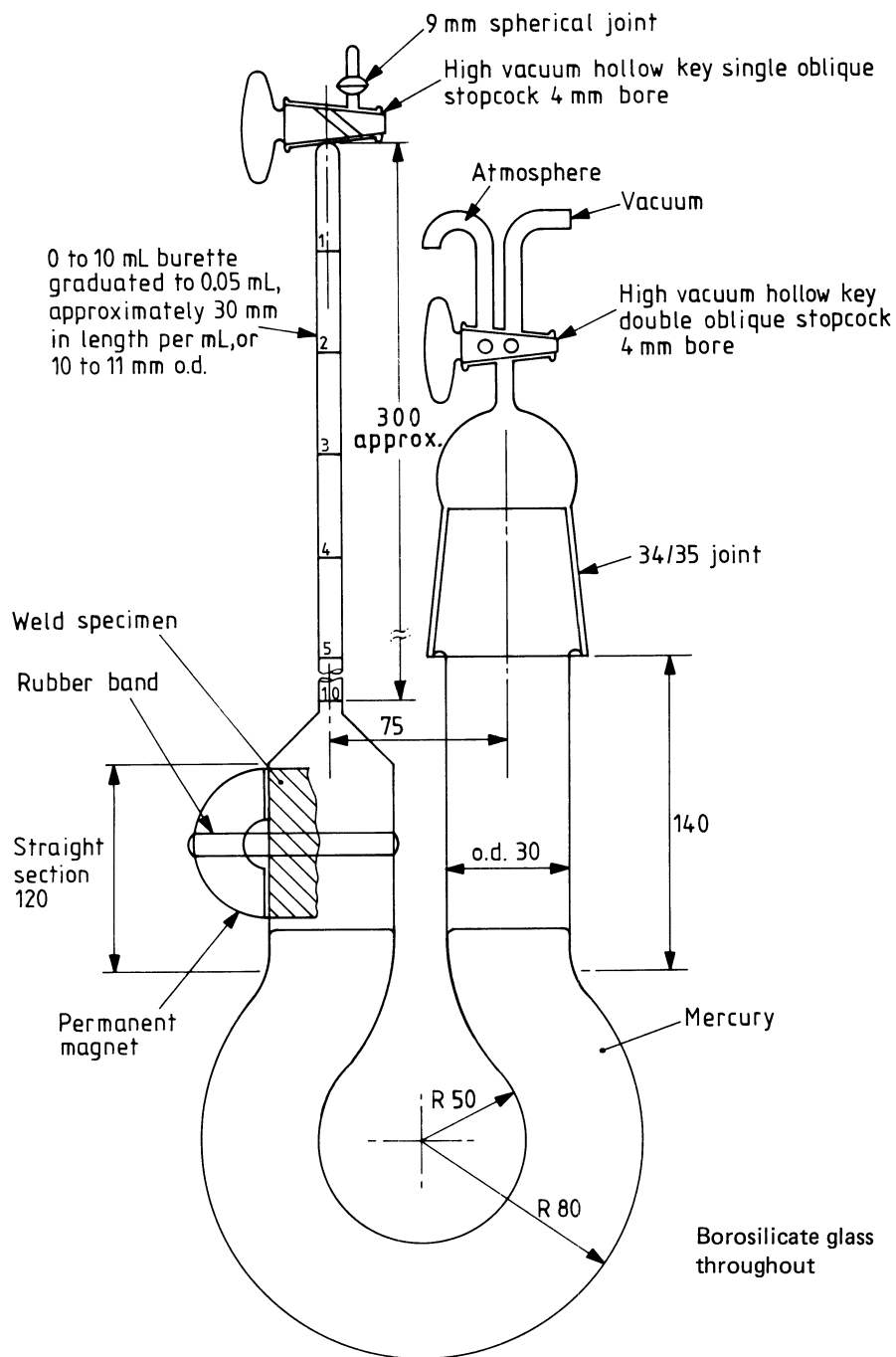
The temperature of the jig shall be 25 ± 5 °C prior to testing. The welding current shall be the maximum stated by the manufacturer less 15 A, the machine setting being controlled within a tolerance of ± 5 A. The traverse speed of the electrode shall be between 12 mm and 13 mm per 10 mm of bead length. The time spent in the deposition shall be noted.

3 s after extinction of the arc, the jig shall be released and the test piece assembly shall be quenched in iced water and subsequently in alcohol or acetone saturated with solid carbon dioxide. The sample pieces shall be wire brushed and broken apart while cold, using brief intermittent periods of cooling; the intervals spent outside the cooling bath in these operations shall not exceed 10 s each. The samples may now be stored at solid carbon dioxide temperature for a period up to 3 days before analysis.



All dimensions are in millimetres.

Figure 1 — Test piece assembly for hydrogen sampling of weld deposits



All linear dimensions are in millimetres.

Figure 2 — Example of diffusible hydrogen collecting apparatus

4.3 Preparation of sample for analysis

When transferring the samples to the gas burette, a shield of dry nitrogen shall be applied to avoid condensation of atmospheric humidity. The sequence of operations and the time spent in each shall be as follows.

- a) The sample shall be washed in alcohol for a period between 3 s and 5 s.
- b) The sample shall be washed in pure ether for a period between 3 s and 5 s.
- c) The sample shall be dried in a blast of dry nitrogen supplied from a nozzle, particular attention being paid to the fractured faces of the specimen. This operation shall be accomplished in not less than 20 s and not more than 22 s.
- d) While maintaining a blanket of dry nitrogen, the sample shall be transferred to the outer limb of the burette, where the sample shall be held in position clear of the mercury surface by a magnet.

The outer limb of the burette shall then be evacuated down to a pressure of approximately 0.1 mmHg¹⁾.

The time spent in these operations shall be not less than 20 s and not more than 25 s.

- e) The sample shall be transferred through the mercury air-lock to its final position in the measuring limb of the burette. This operation shall be accomplished within 5 s.

The total time spent in transferring the sample until the measurements commence shall not exceed 60 s.

4.4 Analytical procedure

The sample shall be maintained under reduced pressure at 25 ± 5 °C (298 ± 5 K) for a period of 72 h when the final volume shall be measured. The barometric pressure shall be recorded. The sample shall be removed from the apparatus, thoroughly brushed so as to remove any oxide skin, and weighed to the nearest 10 mg.

5 Calculation and expression of results

The volume V_h (in mL) at s.t.p. of diffusible hydrogen per 100 g of deposited weld metal shall be calculated from the following formula²⁾:

$$V_h = \frac{V_g (B - H)}{760} \times \frac{273}{298} \times \frac{100}{(M_2 - M_1)}$$

where

V_g is the volume of gas in the burette after 72 h (in mL);

B is the barometric pressure (in mmHg)¹⁾;

H is the head of mercury at which V_g is measured (in mmHg);

M_2 is the mass of the sample after removal from the apparatus (in g);

M_1 is the mass of the sample before deposition of the weld (in g).

For the purposes of this standard, the average value of the results from the four determinations shall be used (two test piece assemblies with two samples from each).

¹⁾ 1 mmHg = 133.322 Pa (approximately).

²⁾ To convert to parts per million, multiply by 0.9.

Publications referred to

BS 639, *Covered electrodes for the manual metal-arc welding of carbon and carbon manganese steels*³⁾.

BS 5135, *Specification for process of arc welding of carbon and carbon manganese steels*³⁾.

BS 6693, *Diffusible hydrogen*³⁾.

BS 6693-2, *Method for determination of hydrogen in manual metal-arc weld metal*.

ISO 3690, *Welding — Determination of hydrogen in deposited weld metal arising from the use of covered electrodes for welding mild and low alloy steels*³⁾.

³⁾ Referred to in the foreword only.

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