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BRITISH STANDARD

**BS 6693-3:
1988**

*Incorporating
Amendment No. 1*

Diffusible hydrogen —

Part 3: Primary method for the determination of diffusible hydrogen in manual metal-arc ferritic steel weld metal

UDC 621.791.753:621.791.042.4:546.11 – 13.06

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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
British Steel Industry (Wire Section)
Electricity Supply Industry in England and Wales
Engineering Equipment and Materials Users' Association
Process Plant Association
Society of Motor Manufacturers and Traders Limited
Water-tube Boilermakers' Association
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The following body was also represented in the drafting of the standard, through subcommittees and panels:

Aluminium Federation

This British Standard, having been prepared under the direction of the Welding Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 August 1988

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Amd. No.	Date of issue	Comments
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Foreword

BS 6693 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.

Parts 1 and 2 of this standard have been published as they were required by other standards. However they do not contain all the requirements of a primary method. This Part of BS 6693 is based on Part 2. It is anticipated that when Part 3 is fully established and called up in other standards, it will be possible to withdraw both Part 1 and Part 2, particularly as Part 3 makes provision for 72 h diffusible hydrogen measurement.

Because instrument methods are widely used for speed of application, an appendix has been included on relating instrument methods to the primary method given in this Part of BS 6693.

It has been assumed in the drafting of this Part of BS 6693 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.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, 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.

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1 Scope

This Part of BS 6693 describes a method for the determination of diffusible hydrogen in manual metal arc ferritic steel weld metal and where collection of hydrogen is continued until there is no increase in volume on successive days. Provision is made for the measurement of diffusible hydrogen after 72 h collection time. An upper limit of energy input of 2 kJ/mm is imposed by the sample size.

NOTE 1 This Part of BS 6693 is not a classification document, but the method may be used for classification purposes when required.

NOTE 2 The titles of the publications referred to in this Part of BS 6693 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 volumetrically.

NOTE A video recording of the method entitled "Measurement of diffusible hydrogen in test welds" is available from the Welding Institute and is a useful training aid.

3 Reproducibility

Information on the reproducibility of the method is given in Appendix A.

4 Materials and apparatus

4.1 *Test piece assembly*

The test piece assembly shall be prepared from a plain carbon non-rimming steel containing not more than 0.20 % C and not more than 0.06 % S.

4.2 *Electrodes*

The covered electrode to be tested shall be used in one of the following ways.

a) For the purposes of classification, when the electrode and the method of deposition of the weld shall be as specified in the standard with which the electrode complies.

b) For the purposes of investigation, when the electrode and welding parameters shall be those given in the specific welding procedure.

When a predrying treatment is specified, then the time and temperature specified by the consumable manufacturer shall be used.

Electrodes with cracked or broken coatings shall not be used and electrodes to be tested in the as-received condition shall be taken from a freshly opened undamaged packet. During any drying treatment the electrodes shall not touch each other or the side of the oven. During any drying operation the electrodes shall spend the full specified time at the drying temperature as measured by a thermocouple in contact with an electrode. Only electrodes under test shall be placed in the oven during this time. When the drying operation is complete, the electrode shall be used immediately. Any electrodes removed from the drying oven and not then used shall not be redried and subsequently used for the test.

NOTE The arc energy for making the weld is restricted to a maximum of 2 kJ/mm.

4.3 *Welding fixture*

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

NOTE A faster through-put of samples can be achieved by incorporating water cooling channels in the copper fixture.

4.4 *Copper foil*

Annealed copper foil strips 1 mm thick shall be used as inserts between the test piece assembly and welding fixture (see Figure 1).

NOTE The foil may be annealed repeatedly at approximately 700 °C and quenched in water but should only be used once after each anneal. Oxide scale after annealing is removed by pickling with dilute nitric acid (10 %) followed by washing with distilled water and drying.

4.5 *Apparatus for determination of diffusible hydrogen*

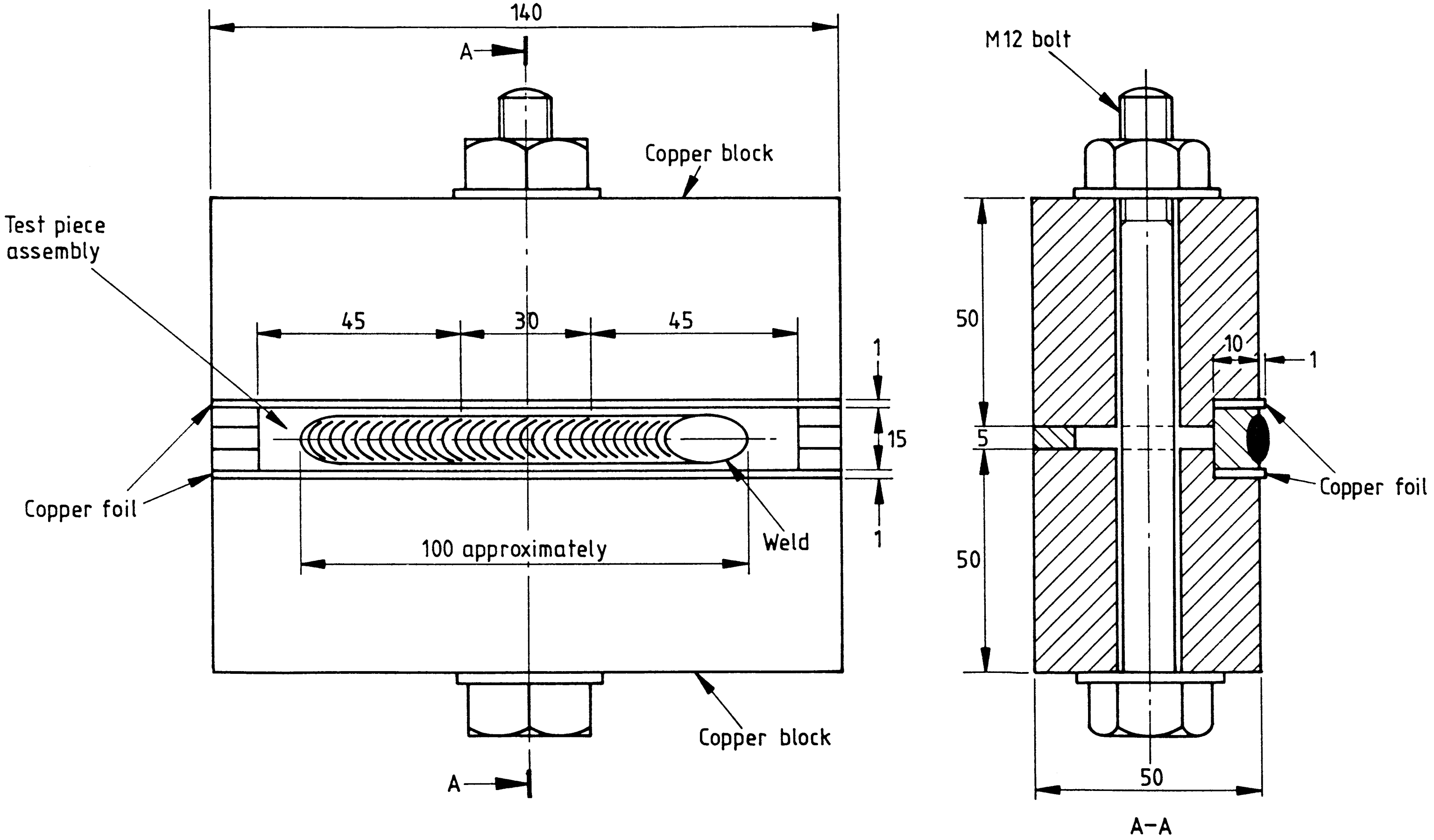
An example of a gas burette for the measurement of extracted gas is shown in Figure 2. Burettes of other designs may be employed, provided that the following requirements are fulfilled.

a) Clean 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 in **5.3.2**, to remove any trace of wash liquid or moisture trapped on the fractured surface of the sample.

NOTE This may be achieved in burettes consisting of a sealed limb, as shown in Figure 2, through manipulation of the mercury level and the stopcock, any contaminants released during the brief period of surface degassing being swept out of the burette before the measurements are taken.

c) A cathetometer shall be used to measure to an accuracy of 0.02 mm the length of gas column in the precision bore limb.



All dimensions are in millimetres.

NOTE Water cooling channels may be used.

Figure 1 — Welding fixture and test piece assembly

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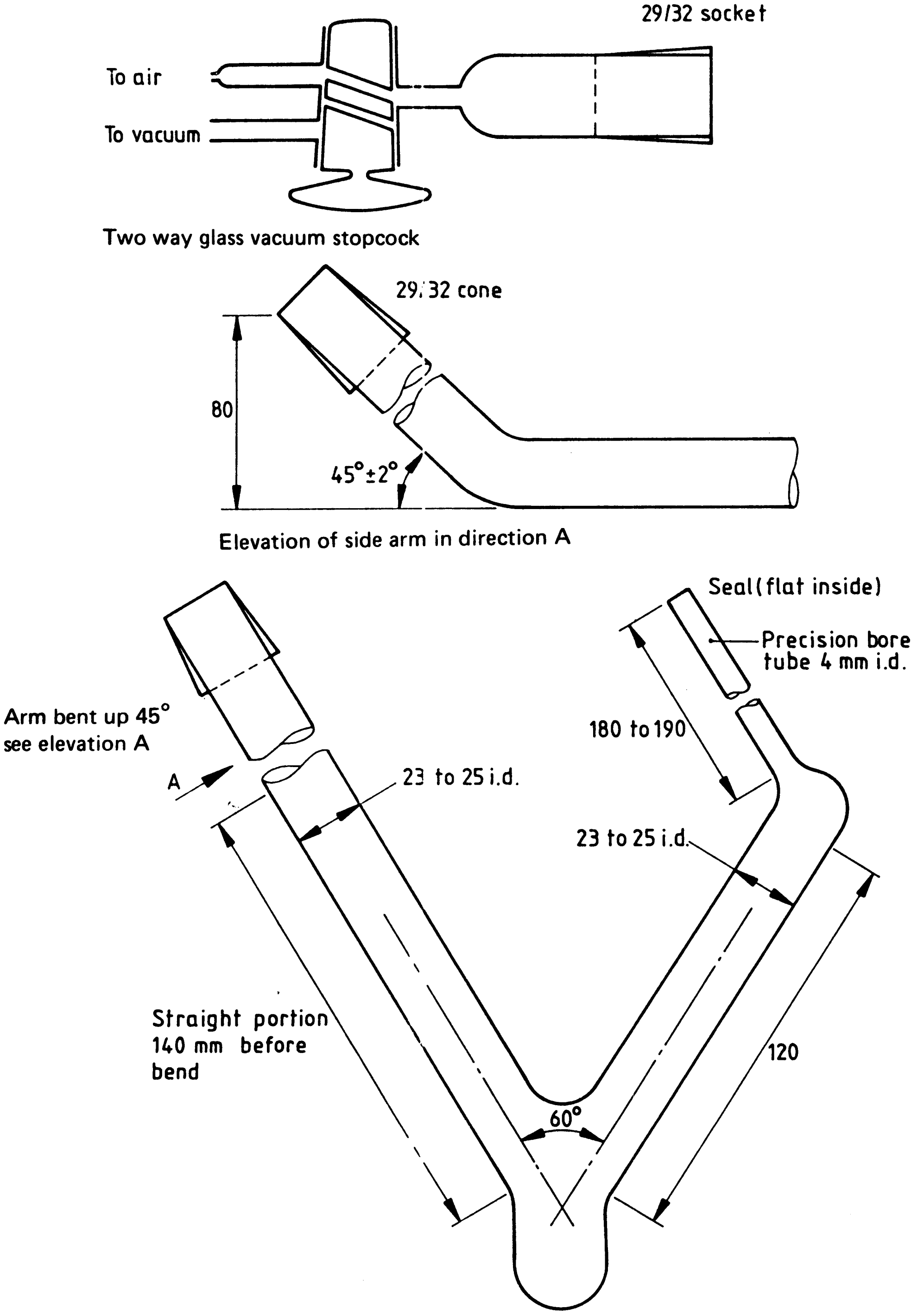

Not to scale. All linear dimensions are in millimetres.

Figure 2 — Diffusible hydrogen collecting apparatus, "Y" tube

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5 Procedure

5.1 Test piece assembly

The test piece assembly shown in Figure 1 consists of 45 mm long run-on and run-off pieces, and a central sample piece of 30 mm length. Triplicate test piece assemblies having a cross section of 10 mm by 15 mm shall be used for each test condition. 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 surface grinding so as to ensure a uniform size. The faces of the cross sections shall be machined to ensure good contact between adjacent pieces. The central sample section shall be marked on the opposite side to that used for welding and weighed to the nearest 10 mg, M_1 (in g). Prior to use, the test piece shall be degassed at 650 °C for 1 h in an inert atmosphere or in vacuum sufficient to prevent oxidation.

5.2 Welding

The test piece assembly shall be clamped in the fixture using annealed copper foil as shown in Figure 1. A torque wrench set at 35 N·m minimum shall be used to ensure that the pressure applied is uniform from test to test.

The temperature of the fixture shall be ambient or not more than 5 °C above ambient prior to testing.

A bead of approximately 100 mm overall length shall be deposited along the centreline of each of the three test piece assemblies. An unused electrode shall be used for each weld. The length of electrode used per unit weld length together with the diameter of the electrodes shall be noted.

The welding current, polarity, voltage, time, weld length and electrode length shall be noted for each of the triplicate welds.

After extinction of the arc and without any delay, the fixture shall be released and the test piece assembly shall be quenched in cold water and quickly transferred to a container of alcohol or acetone saturated with solid carbon dioxide, or of liquid nitrogen. The central sample piece shall be cleaned by vigorous brushing using a steel wire brush in good condition, with brief intermittent periods of cooling or the sample shall be cleaned by grit blasting (see **6.3**); the intervals spent outside the cooling bath during this operation shall not exceed 15 s. The test piece assembly shall then be broken apart and the centre sample section stored in solid carbon dioxide or liquid nitrogen until required for analysis.

5.3 Analysis of sample

5.3.1 *Preparation of gas burette.* The volume of mercury required to fill the size of gas burette illustrated in Figure 2 is about 110 mL. It is essential that the mercury is 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. The gas burette shall be slowly raised to the vertical position and air admitted to the wide limb by rotating the stopcock. Confirm that there is no air bubble at the top of the capillary. If air is present, then the evacuation process shall be repeated until a final check is able to confirm the absence of air. The vacuum stopcock shall be removed from the wide limb of the gas burette.

5.3.2 *Sample handling.* The sample shall be removed from the storage coolant and raised to room temperature.

NOTE 1 The sample can be raised conveniently to room temperature by immersing in water until the ice just melts.

Following a rapid rinse with acetone and drying in a jet of air, the sample shall be transferred to the wide limb of the burette. This shall then be closed with the two-way vacuum stopcock and evacuated. Acetone and traces of condensed water evaporate off the surface of the sample and are removed with the evacuated air. The sample shall be manoeuvred with a magnet into position under the capillary by carefully lowering the burette towards the horizontal position until it just floats along on the mercury surface. It is essential that care is taken to prevent air entering the capillary 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, for example, with a cork or a glass cap, to prevent release of traces of mercury vapour.

NOTE 2 Mercury vapour is emitted from open mercury surfaces at room temperature, therefore accidental spillage should be removed. Mercury can be handled safely on a bench with a raised edge in a well ventilated area.

Diffusible hydrogen is evolved from the sample and collects in the precision bore tube. The whole procedure described in **5.3.2** shall be carried out as quickly as possible.

NOTE 3 This should not take more than approximately 2 min.

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5.4 Analytical procedure

The sample shall be maintained at 25 ± 5 °C until there is no increase in calculated hydrogen on successive days. The length of the hydrogen gas column C and the head of the mercury H shall be measured in millimetres using a cathetometer and room temperature T (in °C) and barometric pressure P (in mm) shall be recorded. The volume of hydrogen collected V (in mL), corrected to standard temperature and pressure, is given by the following equation:

$$V = \frac{273\,(P - H)\,(\pi r^2 C)}{760\,(273 + T) \times 1000}$$

where

r is the radius of the standard bore tube (in mm).

When evolution has ceased, the sample shall be removed from the apparatus and weighed to the nearest 10 mg, M_2 (in g). All the relevant data shall be recorded.

NOTE An example of a form (see **B.1**) for the recording of analysis data is given in Appendix B.

6 Calculation and expression of results

6.1 Diffusible hydrogen in deposited weld metal H_D

The volume H_D (in mL) at STP of diffusible hydrogen per 100 g of deposited weld metal shall be calculated from the following equation:

$$H_D = V \times \frac{100}{(M_2 - M_1)} \text{ mL per 100 g of deposited weld metal}$$

6.2 Diffusible hydrogen in fused metal H_F

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 the deposited metal. These shall be measured on the ends of the sample by using an enlarged tracing or photograph, or an image analysing microscope and averaging the results as shown by the following equation:

$$H_F = H_D \times 0.892 \times \frac{\text{Average area of deposited weld metal}}{\text{Average area of fused metal}} \left(\text{parts per million by mass}\right)$$

6.3 72 h diffusible hydrogen measurement

The analytical procedure described in **5.4** shall be used to provide the diffusible hydrogen fraction which has been evolved after a 72 h collection time. To optimize hydrogen evolution, grit blasting with alumina shall be used to clean the sample surface when using 72 h collection.

6.4 Reporting of results

All data which can be relevant to the interpretation of results shall be reported. For the purposes of this Part of BS 6693, the average value of the hydrogen concentration of triplicate welds shall be reported.

NOTE An example of a report form (see **B.2**) is given in Appendix B.

7 Instrument methods

The use of instrument methods is considered in Appendix C which gives guidelines on the correlation of the results from rapid methods with those obtained by the primary method given in this Part of BS 6693.

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Appendix A Reproducibility of the method

The method of obtaining standard deviations is given in BS 4237, and for the purposes of planned analytical trials, any standard deviation is obtained from the equation:

$$S = \sqrt{S_b^2 + S_w^2}$$

where

S_b is the between-operator standard deviation;

S_w is the within-operator standard deviation.

A planned trial[1] of this procedure was carried out by six analysts, from five laboratories, six tests being performed by each analyst. From the results obtained, the 95 % confidence limits (2S) given in Table 1 were calculated.

The reproducibility index is reduced by a factor of $\frac{1}{\sqrt{3}}$ when the mean of triplicate results is reported.

Table 1 — Laboratory tests' confidence limits

	Diffusible hydrogen	**Components of standard deviation**		**Reproducibility index for a single result**
		S_b	S_w	2S
Complete, deposited weld metal (H_D) in mL/100 g	9.6	0.355	0.797	1.74
Complete, fused weld (H_F) p.p.m.	4.5	0.279	0.547	1.23
72 h deposited weld	8.2	1.052	0.812	2.66

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[1] "The measurement of diffusible hydrogen in manual metal-arc weld metal deposits". A study group report, International Institute of Welding document II-A-576-82.

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Appendix B Recording of data

B.1 Analysis data sheet (diffusible hydrogen, manual metal arc)

All data required for the calculation of the diffusible hydrogen may be recorded as follows:

Date:

Hydrogen collection temperature: °C

Hydrogen collection time: d h

Barometric pressure
(recorded during measurement) (P): mmHg

Room temperature
(recorded during measurement) (T): °C

Radius of capillary: mm

	1	2	3
Mass of centre test pieces (M_1): g:			
Mass of centre test piece plus weld (M_2): g:			
Mass of deposited weld metal ($M_2 - M_1$): g:			
Average area of deposited metal: mm^2:			
Average area of fused metal: mm^2:			
Length of hydrogen gas column (C): mm:			
Head of mercury in capillary (H): mm:			

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B.2 Report form (diffusible hydrogen, manual metal arc)

The following report sheet gives full details of all the test variables which pertain to the test results.

Investigating laboratory: Date:

Investigator's name:

Make of electrode: Batch No.:

Type of electrode: Electrode designation:

Diameter of electrode mm: Overall length of electrode: mm:

Drying treatment: °C for h:

Electrode polarity DC +ve, DC –ve or AC:

Relative humidity (%) and temperature (°C) during welding:

Approximate evolution temperature: °C

Hydrogen collection time: d h

	1	2	3
Voltage V: AC or DC:			
Current A: type of meter:			
Welding time: s:			
Weld length: mm:			
Energy input: kJ/mm:			
Electrode length used: mm:			
Runout ratio:			
Mass of weld metal deposited on test piece. g:			

Diffusible hydrogen	1	2	3	Average	
(a) H_D, deposited weld					mL/100 g
(b) H_F, fused weld					p.p.m.

Other test details not included above:

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Appendix C Rapid methods for the measurement of diffusible hydrogen in ferritic arc weld metal

The primary method for the measurement of diffusible hydrogen in ferritic arc weld metal is based upon collection and measurement over mercury of the hydrogen evolved from a standard-sized weld sample. The evolution takes place at room temperature and consequently the collection time is typically about 14 days. This time-scale is acceptable in a primary method, but when results are required for purposes such as quality control, or release of a consumable for sale, then more rapid techniques of measurement are required.

In order to reduce the hydrogen evolution time, it is necessary to heat the weld sample and the mercury method cannot therefore be used. The heating temperature will determine the time taken for complete evolution to occur. For example, a temperature of 45 °C is said to enable analysis to be carried out in 3 days. The higher the temperature the quicker the evolution and at 650 °C analysis for total hydrogen can be achieved within 30 min. The choice of temperature is important because above about 150 °C there will be significant dissociation, and release, of hydrogen which at room temperature would remain in the molecular state and be permanently trapped in voids in the weld metal. 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.

It is not the objective of this appendix to describe the several commercial instruments which are available for the measurement of hydrogen in metals. However it is important to note that any instrument incorporating the facility for measuring diffusible hydrogen in weld metal has to produce results which are equated, in terms of accuracy and reproducibility, with room temperature diffusible hydrogen results obtained using the primary method presented in this Part of BS 6693.

When evaluating the suitability of an instrument for the measurement of diffusible hydrogen in standard test welds, it is essential that the following factors are examined.

a) *Calibration*

Calibration of analytical instruments is normally achieved by using certified reference materials to quantify instrument response. Further, it is normal to check calibrations by performing regular analyses of reference materials or secondary standards. In the case of hydrogen, such reference materials and secondary standards are not available because of the transitory mode of occurrence of hydrogen at, or above, room temperature. Calibration by injection of pure hydrogen is, in practice, not always the primary method it appears to be. This is because of the difficulty of reproducing the characteristics of hydrogen evolution from a weld sample with the dynamic gas flow systems of the instrumental methods.

Because standard samples are not available, calibration has to be by injection of known volumes of hydrogen into the carrier gas system over the operating range of interest.

The instrument should first be set up according to the manufacturer's instructions and left to stabilize for a sufficient period of time with the carrier gas flowing. This period could be up to 2 h. Calibration is then performed by injecting hydrogen volumes ranging from 0.05 mL to 0.95 mL at STP in steps of 0.10 mL at STP. This corresponds to a range of deposited weld metal contents of from 1.5 mL to 30 mL at STP per 100 g for a typical manual metal arc deposit mass of approximately 3.5 g obtained using the primary method. Higher deposit masses, arising when testing submerged arc consumables for example, may give volumes of hydrogen beyond this range. In those circumstances the calibration should be extended accordingly.

During normal instrument use, any existing calibration should be checked by gas dosing before analysis is attempted. The prior stabilizing procedure should also be adhered to. Checks should be made at hourly intervals during a series of analyses to ensure that no drift has occurred in the instrument calibration.

b) *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 coefficient r. Values of r close to unity indicate a high degree of correlation.

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The range of hydrogen contents to be measured will range from 0.05 mL to over 1.0 mL at STP. Hydrogen, injection should cover this range to confirm linearity of response, but tests with weld specimens should be carried out to confirm that the hydrogen evolution characteristics of the sample are followed in a linear fashion by the instrument.

c) *Accuracy*

There are no primary standards available which will release diffusible hydrogen at temperatures below 150 °C. The only way in which accuracy may be assessed is by a statistical comparison of a series of nominally identical test welds, divided into two groups in a random fashion, and the first group being analysed by the instrument, whilst the second group is analysed by the primary method.

The accuracy of the instrument should be determined at several levels of hydrogen content. These should include the 5 mL, 10 mL and 15 mL at STP per 100 g deposited weld metal control levels referred to in BS 639 and BS 5135. A further check at the 25 mL level should be done to cover the analysis of non-hydrogen controlled consumables.

It is recommended that nine repeat determinations are carried out using both the rapid method and the primary method described in this Part of BS 6693. Samples should be representative of the consumables to be tested. Any variations between individual electrodes will, of course, contribute to the standard deviations obtained. The accuracy of the rapid method is then judged by assessing the statistical significance of the difference in means of the two sets of results. Following BS 4237, if the probability of the difference not being due to chance is greater than 95 %, then the difference in means is probably significant. The most common statistic to use when comparing means is the t value defined as shown in the following equation:

$$t = \frac{\text{Difference of means}}{\text{Standard error of difference}}$$

The following equation may be used to calculate t:

$$t = \frac{|\bar{x}_R - \bar{x}_P|}{\sqrt{\frac{S_R^2}{n_R} + \frac{S_P^2}{n_P}}}$$

where

x_R, S_R, n_R and x_P, S_P, n_P are the mean, standard deviation and number in sample for the rapid and primary method respectively.

The t value so obtained is referred to tables of statistics where, for the number of degrees of freedom involve $(n_P + n_R) - 2$, the probability of this value having arisen by chance will be given. If the difference in means is judged to have arisen by chance, then the two methods may be assumed to give identical results.

d) *Reproducibility*

A series of repeat welds, analysed as indicated for the tests on accuracy, will also provide information on the reproducibility of the instrumental method.

Reproducibility is the consistency of replicate tests and is expressed by the standard deviation S. A reproducibility index, $2S$, may be defined and in statistical terms 95 % of results would lie within a band $\bar{x} \pm 2S$, where $\bar{x}$ is the mean value. A decrease in the numerical value of S implies an increase in reproducibility.

Reproducibility of a method (see Appendix A) is best determined using a planned trial in which analysts from several laboratories are involved so as to characterize the within-operator and between-operator components of the standard deviation given by the following equation:

$$\bar{S} = \sqrt{S_b^2 + S_w^2}$$

where

S_b is the between-operator standard deviation;

S_w is the within-operator standard deviation.

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The standard deviation of the nine results of each of the control levels mentioned under item c) "Accuracy" gives values of S_w for both the rapid and primary methods.

e) *Blank*

A blank should be carried out to determine the instrument response to a standard sized degassed specimen. This operation is advisable on a regular basis to confirm proper functioning of the instrument.

It should be noted that the instrument response, as shown by the increments of the readout, has an influence upon both the accuracy and the reproducibility. For example, a readout of 0.01 mL at STP represents 0.25 mL H_2 per 100 g of deposited weld, or 0.12 p.p.m. H_2 per 100 g of fused weld.

Prior to each analytical session, to confirm proper functioning of the instrument, a weld deposit produced according to this Part of BS 6693 should be analysed. This deposit should have been previously outgassed for 1 h at 650 °C under vacuum or inert gas atmosphere to ensure that it does not contain diffusible hydrogen. If the rapid method employs a higher analysis temperature than 650 °C, then the higher temperature should be used for prior outgassing of the sample blank.

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Publications referred to

BS 639, *Specification for covered carbon and carbon manganese steel electrodes for manual metal-arc welding.*

BS 4237, *Report on reproducibility of methods of chemical analysis used in the iron and steel industry.*

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

BS 6693, *Diffusible hydrogen*[2].

BS 6693-1, *Method for determination of hydrogen in manual metal arc weld metal using 3 day collection.*

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

Measurement of diffusible hydrogen in test welds (video recording)[3].

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[2] Referred to in the foreword only.

[3] Available from the Welding Institute, Abington Hall, Abington, Cambridge CB1 6AL.

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