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भारतीय मानक ब्यूरो

DRAFT FOR WC

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भारतीय मानक प्रारूप

ऑस्टेनाइटीक स्टेनलेस इस्पात की अंतकणिका संक्षारण के प्रतिरोध की निर्धारण विधि

भाग १ द्रव्यमान में हानि के मापन द्वारा नाइट्रिक एसिड माध्यम में संक्षारण परीक्षण (हुए परिक्षण)

(IS 10461 (भाग 1) का दूसरा पुनरीक्षण)

Draft Indian Standard

Resistance to Inter-Granular Corrosion of Austenitic Stainless Steels — Method for Determination

Part 1 Corrosion Test in Nitric Acid Medium by Measurement of Loss in Mass (Huey Test)

(Second Revision of IS 10461 (Part 1))

ICS 77.060

Corrosion Protection and Finishes Sectional Committee, MTD 24	Last date for receipt of comments is **22 November 2024**

FOREWORD

(*Formal foreword clause will be added later*)

Intergranular corrosion denotes deterioration of metals by means of preferential attacking of the grain boundaries. For austenitic stainless steels, which may be subject to such as attack when they are kept at a temperature between about 430 ºC to 820 ºC, it is associated with the precipitation of chromium rich carbides or intermetallic compounds, such as sigma phase at grain boundaries. The heat cycle, which may provoke sensitization to intergranular corrosion, may occur during hot-forming process like forging and rolling, as the result of incorrect solution treatment or during a welding operation.

This standard (Part 1) was first published in 1983 providing a uniform and rationalized method of determining the susceptibility of austenitic stainless steels to intergranular corrosion. While formulating this standard considerable assistance has been taken from ISO 3651-1 : 1998 Austenitic Stainless Steels - Determination of resistance to intergranular corrosion - Part 1 : Corrosion test in nitric acid medium by measurement of loss in mass (Huey Test), issued by the International Organization for Standardization.

This revision has been brought out to bring the standard in the latest style and format of the Indian Standards. In addition, following modification have been done:

a) Note has been added in scope; (**1**)
b) Fig 1 has been updated; (**Fig.1**)
c) Requirements of corrosive solution has been modified;(**4.2**)
d) Clause on chemical preparation has been modified;(**4.4.2.2**) and
e) Test report clause has been added; (**5**)

This standard is published in two parts. The other part in the series under the general title 'Resistance to Inter-Granular Corrosion of Austenitic Stainless Steels — Method for Determination' is:

Part 2 Corrosion test in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss Test)

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Draft Indian Standard

RESISTANCE TO INTER-GRANULAR CORROSION OF AUSTENITIC STAINLESS STEELS – METHOD FOR DETERMINATION

PART 1 CORROSION TEST IN NITRIC ACID MEDIUM BY MEASUREMENT OF LOSS IN MASS (HUEY TEST)

(Second Revision)

1 SCOPE

1.1 This standard describes a procedure for determination of the resistance to intergranular corrosion of austenitic stainless steels in a nitric acid medium by measurement of the loss in mass, (Huey Test).

1.2 This method is applicable to austenitic stainless steels supplied in the form of castings, rolled or forged products and tubes including weld metals and intended to be used in an oxidising media (for example, relatively concentrated nitric acid). In general, the Huey test should not be used for grades containing molybdenum unless the material tested is to be used in nitric acid service.

1.2.1 The presence or absence of intergranular corrosion in these tests is not necessarily a measure of the performance of the material in, other corrosive media. The tests do not provide a basis for predicting the resistance to other forms of corrosion such as general corrosion, pitting or stress corrosion cracking.

NOTE – It is important to note that the result of the corrosion test is only strictly valid for the corrosive medium used in the test. It constitutes a basis for estimating the resistance to intergranular corrosion but may not be used to check resistance to other forms of corrosion (general corrosion, pitting, stress corrosion, etc.). It is necessary for the user to adapt the specified corrosion test to the use which will be made of the alloy. This test should, in no case, be considered as an absolute criterion of the quality of the alloy.

2 PURPOSE OF THE TEST

2.1 The test indicates susceptibility to intergranular attack associated with the precipitation of, chromium rich carbides or intermetallic compounds such as sigma phase at the grain boundary. Some type of inclusions (active inclusions) are also susceptible to attack in this test.

2.2 This test may be used to evaluate any of the following three factors:

a) The effectiveness of the final treatment when the material is tested in the as delivered condition without further test heat treatment sensitization;
b) The effectiveness of the added stabilizing elements, or low carbon content when the material is tested in the solution heat treated and subsequently sensitized condition; and
c) The influence of welding and associated post fabrication/post weld heat treatments.

3 HEAT TREATMENT FOR SENSITIZATION

In order to verify the effectiveness of the added stabilizing elements or low carbon content ($C \leq 0.03\%$) to resisting intergranular corrosion, it is necessary to carry out a heat treatment for sensitization. This treatment is usually obtained by maintaining a test pieces for 1 h at a temperature of 650 °C to 675 °C followed by rapid cooling (in water) to room temperature in air or water unless other specific heating or cooling rates are stipulated. The duration of the rinse in temperature shall not exceed 10 min. For low carbon variety of stainless steels, the most commonly used heat treatment is heating at 675 °C for 1 h followed by rapid cooling in air or water. This heat treatment results into appreciably high precipitation of carbides and also simulates to a certain extent the worst microstructure which is obtained after welding in the heat affected zone (HAZ).

4 CORROSION TEST

4.1 Principle

A test piece, prepared as specified in **4.4.2**, is weighed; then immersed in a boiling solution of nitric acid for 5 periods, each of 48 h using fresh solutions for each period. The criterion for evaluating the test is loss in mass determined by weighing after each period.

4.2 Corrosive Solution

The corrosive solution is an aqueous solution of 65 % ± 0.2 % (m/m) mass percent reagent grade nitric acid (r.d. 1.40 g/ml).

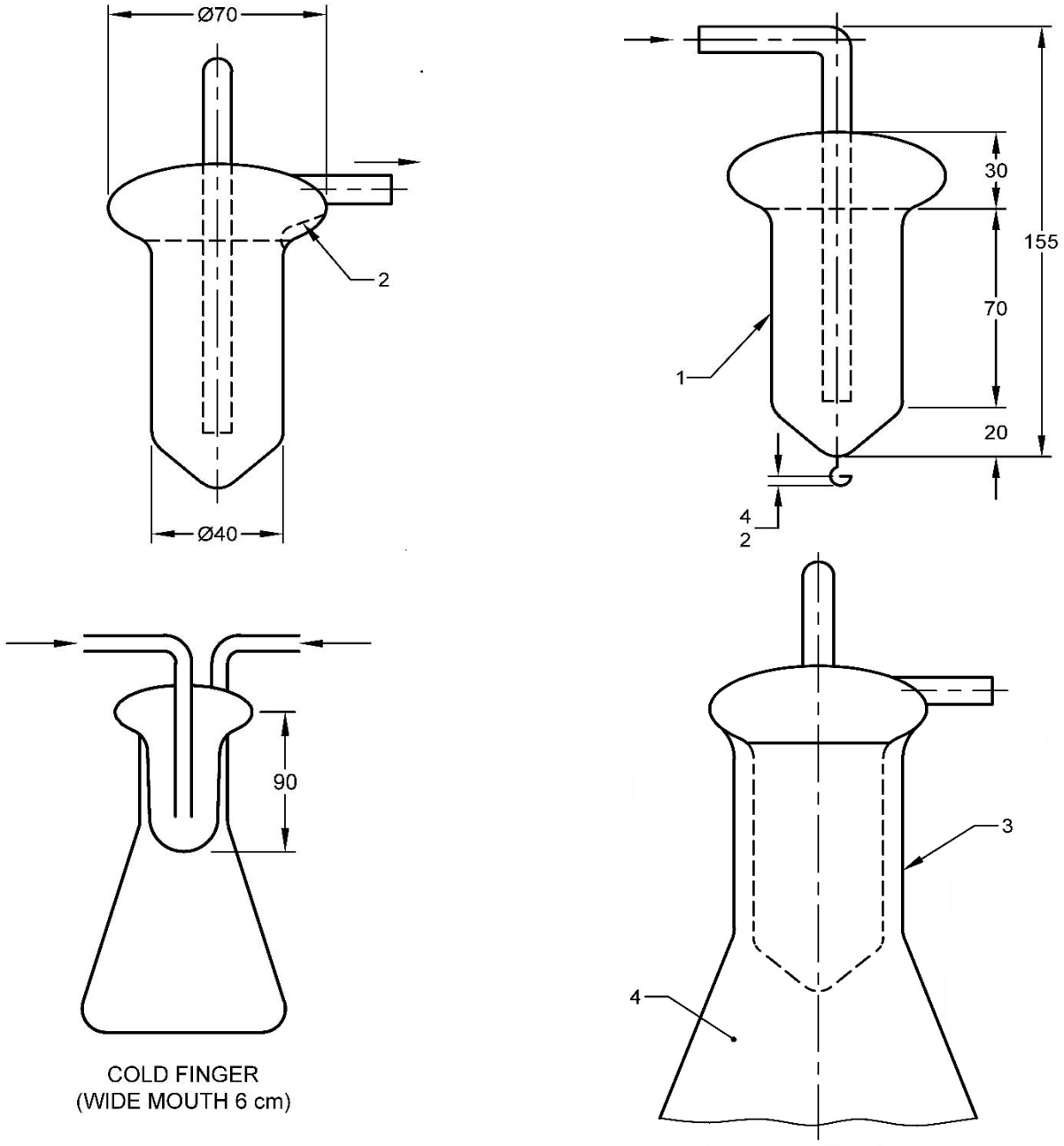
NOTE – The nitric acid conform to the specifications for analytical reagent chemical and shall have the following residual contents:

Fixed residue	≤ 50 mg/kg
Pb	≤ 5 mg/kg
Fe	≤ 2 mg/kg
Mn	Negative test
As	≤ 0.05 mg/kg
Cl^{-1}	≤ 1 mg/kg
SO_4^{-2}	≤ 10 mg/kg
PO_4^{-3}	≤ 2 mg/kg
F^{-1}	≤ 1 mg/kg

4.3 Apparatus

4.3.1 *Conical Flask* **—** of capacity at least 1 litre, fitted with a "cold finger" immersion condenser. The corrosion rates depend on the type of condenser used (*see* Fig. 1). By means of a paper indicator, it shall be checked that no acid fumes are given off during the test. For comparative measurements, the apparatus shall remain identical for all the tests.

NOTE — It has been shown that corrosion rates obtained with a reflux condenser tend to be somewhat higher than with the cold finger type condenser due to greater vapour loss.



All dimension in mm

Key :

1 Thickness of Glass = 1.5mm
2 2 or 3 Notches (to avoid joining by condensation)
3 Monitoring the Scheme of Condenser
4 Erlenmeyer Flask

FIG. 1 TYPES OF COLD FINGER CONDENSERS

4.3.2 *Support for the Test Piece* — generally made of glass

4.3.3 *Heating Device* — to keep the solution boiling.

4.4 Test Piece

4.4.1 Dimensions

The test piece taken from the product shall have its largest dimension located in the direction of working. The test piece dimension shall be determined as a function of the available weighing facilities and the volume of solution to be used. The test piece length shall, however, be at least equal to twice its width, and the total surface area of the section, perpendicular to the direction of working or the fibres, shall be less than ~~50~~ 15 percent of the total surface area of the test piece. In comparative tests, the ratio of the total surface area to the total surface area of the sections shall be kept constant. The welded specimens should be cut in such a way that more than 13 mm width of base metal is included in either side of the weld.

4.4.2 *Preparation*

Depending on the purpose of the test (*see* **2.2**), the test piece, either with or without sensitization treatment, shall be prepared as specified in either **4.4.2.1** or **4.4.2.2**. Unless stated on the order to the contrary, the method of preparation, shall be left to the discretion of the manufacturer.

4.4.2.1 *Mechanical preparation*

The test piece shall be descaled mechanically by polishing on all surfaces, including the edges, with grade 120 or finer, iron-free abrasive paper or cloth. Over-heating of the test piece shall be avoided.

4.4.2.2 *Chemical preparation*

The test piece shall be descaled for no more than 1 h, without any previous mechanical treatment, in a solution consisting of 50 volumes of hydrochloric acid (r.d 1.19 g/ml), 5 volumes of nitric acid (r.d 1.40 g/ml), and 50 volumes of water at 50 °C to 60 °C, or in a solution of 50 volumes of hydrochloric acid and 50 volumes of water at ambient temperature.

Care should be taken to ensure no chemical is left on the surface of specimen after chemical-treatment. The surface should be thoroughly cleaned using soap solution, water and alcohol/acetone for drying.

In the case of the chemical preparation, it shall be necessary to ensure in advance that intergranular corrosion does not occur due to the preparation. This should be achieved by micro examination of samples of each steel grade tested.

4.4.2.3 *Degreasing*

The test piece shall then be degreased, immediately before being placed in the corrosive solution, rinsed and dried using nonchlorinating reagents such as soap and acetone.

NOTE – The preparation treatment described in **4.4.2.1**, may be supplemented by immersing the specimen in nitric acid for example 20 percent (weight) at 50 °C to 60 °C for 20 min followed by degreasing and drying. In case of small diameter tubes which cannot be conveniently mechanically polished on inside, it is suggested to immerse the specimen in boiling nitric acid (65 percent) for 2 to 4 h using the same apparatus as for the actual tests. This treatment removes any surface contamination that may not be possible by regular cleaning and would otherwise result in increase in the apparent weight loss of the specimen during early part of the test.

4.5 Procedure

4.5.1 Use a volume of corrosive solution (*see* **4.2**) of at least 20 ml per square centimetre of surface area of the test piece. In general, only one test piece shall be placed in each conical flask. However, it is possible to test several test pieces at the same time, on condition that they all come from the same grade of steel and are isolated from one another at a distance of at least 5 mm.

4.5.2 Determine the mass of the test piece to an accuracy of 0.001 g as well as its surface area to an accuracy of 5 percent. Then immerse the test piece in the corrosive solution and bring the solution to the boil. Boil for 5 periods of 48 h ± 1 h each, using a fresh solution for each period. After each period, wash the test piece in water, dry and weigh. Care shall be taken to avoid excessive bubbling during boiling.

NOTE – By special agreement between the interested parties, the test may be limited to 3 periods of 48 h each.

4.5.3 In the case of several test pieces being tested in the same flask, the test shall be completely cancelled if, after a period of time, an exaggerated loss in mass of one of the test piece is noted. The test shall be repeated completely on the same test piece which shall be tested separately after having undergone a fresh surface preparation.

4.6 Expression of Results

4.6.1 The effect of the attack by the nitric solution is measured by the mean loss in mass of the specimen per period and for the total of the test periods.

4.6.2 The corrosion rate is given either in millimetres per year (mm y^{-1}) by formula (1) or in grams per square metre per h (g/m^{2}h) by formula (2):

$$r_{corr} = \frac{87\,600\,m}{S \times d \times t} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$r_{corr} = \frac{10\,000\,m}{S \times t} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where

t = period of attack (actual boiling period), in h;

S = original surface area of the test piece, in square centimetres;

m = mean loss in mass per period, in, grams; and

d = density of the test piece (g/cm^3).

4.6.3 *Sample Results*

Stainless steels (Type 304 and 304 L) are often used in concentrated nitric acid environments as in the case of the processing of nuclear fuels, wastes, etc. In such cases, mass loss in the Huey test shall not exceed 0.46 mm y^{-1} (equivalent to 18 mills per year). This may be considered as a general guidance. If the corrosion rate for each period increases over that of the previous period, the material can be considered to be susceptible to intergranular corrosion.

5 TEST REPORT

The test report shall contain at least the following information:

a) reference to this standard;
b) stainless steel grade;
c) heat treatment, if any;
d) type of condenser used;
e) mean corrosion rate; and
f) any incident which may have an effect on results.

Normally the average result for all the test periods is reported. Results from the individual periods may be reported by agreement between the interested parties.