BRITISH STANDARD

BS EN
10229:1998

Evaluation of resistance of steel products to hydrogen induced cracking (HIC)

The European Standard EN 10229:1998 has the status of a British Standard

ICS 77.060

National foreword

This British Standard is the English language version of EN 10229:1998.

The UK participation in its preparation was entrusted to Technical Committee ISE/72, Methods of physical and metallographic testing, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 9 and a back cover

This British Standard, having been prepared under the direction of the Engineering Sector Board, was published under the authority of the Standards Board and comes into effect on 15 July 1998

ISBN 0 580 29798 5

Amendments issued since publication

Amd. No.	Date	Text affected

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 10229

March 1998

ICS 77.060

Descriptors: Tests, cracking tests, estimation, cracking (fracturing), crack propagation, definitions, test specimen, procedure

English version

Evaluation of resistance of steel products to hydrogen induced cracking (HIC)

Evaluation de la résistance des produits en acier à la fissuration induite par l'hydrogène (HIC)

Bewertung der Beständigkeit von Stahlerzeugnissen gegen wasserstoffinduzierte Rißbildung (HIC)

This European Standard was approved by CEN on 2 March 1998.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN 10229:1998 E

Foreword

This European Standard has been prepared by Technical Committee ECISS/TC 1, Steels — Mechanical and physical tests, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1998, and conflicting national standards shall be withdrawn at the latest by September 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Contents

Introduction

The need, today, to drill deeper to find oil and natural gas and the procedures that are being used in many fields to enhance oil and gas recovery are resulting in an increase worldwide in the number of fields considered "sour". This in turn is leading to an increasing demand from oil companies for steels resistant to sour conditions. Flow lines or gathering pipelines in sour fields may be transporting crude oil or natural gas containing significant amounts of hydrogen sulfide (H_2S) and water. Additionally, there is increased recognition of the importance of sour operating conditions in pressure vessels and structural steel work.

On steel, the presence of water with H_2S can cause corrosion. Atomic hydrogen generated by the corrosion reaction can be absorbed into the steel and lead to cracking of the product. Cracks on adjacent planes may link up to form through thickness "steps" and in some instances surface blistering may occur. Hydrogen induced cracking (HIC) occurs without applied stresses.

The test described in this European Standard is not intended to duplicate service conditions, nor show how a material will perform in service. It is an accelerated corrosion test designed as a reproducible procedure capable of evaluating the resistance to hydrogen induced cracking.

NOTE A draft "*Corrosion protection — Carbon and alloy steels for use in H_2S* containing environments in oil and gas production — Materials requirements and test method" being currently prepared by CEN/TC 262, Protection of metallic materials against corrosion, uses also the term "step wise cracking (SWC)".

1 Scope

This standard specifies a method of evaluation of the susceptibility to hydrogen induced cracking (HIC) of steel products with nominal thicknesses equal to or greater than 6 mm.

NOTE This standard may be applied by agreement to products with nominal thicknesses lower than 6 mm.

This standard does not cover resistance to other types of corrosion such as stress corrosion cracking.

2 Definitions

For the purposes of this standard, the following definitions apply.

2.1 sample

a sufficient quantity of material taken from the product for the purpose of producing three test pieces; for example: ring in case of tubes, part of plates

2.2 test piece

part of the sample with specified dimensions, machined for submission to the test

2.3 section

the part which is cut from each test piece after testing, metallographically prepared and examined in order to assess the cracking present. Three sections are taken per test piece

2.4 crack

a more or less planar void discontinuity in the steel

2.5 hydrogen induced crack

a crack below and approximately parallel to the surface of the product, initiated and propagated by the action of hydrogen in the steel as a result of contact with a wet sour medium

2.6

separation between cracks

the shortest straight line distance between two cracks

2.7 isolated crack

a crack separated from the next crack by more than 0,50 mm, with a minimum length equal to or greater than 0,1 mm (see Figures 1 and 2)

2.8 crack system

a combination of two or more cracks, each of which is within 0,50 mm of the next crack (see Figures 1 and 2)

2.9 extent of cracking

the magnitudes of the longitudinal and transverse components of a crack or crack system are referred to as "the longitudinal extent of cracking E_{LC}" and "the transverse extent of cracking E_{TC}", respectively (see Figure 1).

NOTE All hydrogen induced crack systems have longitudinal and transverse components.

2.10 stepwise crack

crack system in which the transverse component is equal to or greater than 0,1 mm

3 Principle of the method

The method consists of exposing test pieces without any applied stress to a corrosive medium for a period of 96 h, followed by evaluation of the test pieces. The corrosive medium is a H_2S saturated solution which may be either an acidified sodium chloride solution (test solution A; see **4.1**) or a synthetic seawater (test solution B; see **4.2**). Other intermediate solutions may also be agreed between purchaser and supplier.

4 Test solutions

4.1 Test solution A

The acidified sodium chloride solution shall be prepared by dissolving 50 g of sodium chloride (NaCl) and 5 g of glacial acetic acid (CH_3COOH) in 945 ml of water. The initial pH shall be 2,7 ± 0,1 before H_2S is introduced.

4.2 Test solution B (synthetic seawater)

To prepare 10,0 l of synthetic seawater, dissolve 245,34 g of sodium chloride (NaCl) and 40,94 g of anhydrous sodium sulfate (Na_2SO_4) in 8 l to 9 l of water. Add slowly with vigorous stirring, 200 ml of solution B1 and then 100 ml of solution B2. Dilute to 10,0 l with distilled or deionized water. Adjust the pH to 8,2 ± 0,1 with 0,1 M sodium hydroxide (NaOH) solution or 0,1 M hydrochloric acid (HCl) before H_2S is introduced (see note).

NOTE Only a few millilitres of NaOH solution should be required.

a) *Solution B1*

The solution shall be prepared by dissolving the indicated amounts of the following salts in water and dilute to a total volume of 7,0 l:

— $MgCl_2 \cdot 6H_20$: 3 889,0 g (= 555,6 g/l);

— $CaCl_2$ (anhydrous): 405,6 g (= 57,9 g/l);

— $SrCl_2 \cdot 6H_20$: 14,8 g (= 2,1 g/l).

The solution shall be stored in well-stoppered glass or other chemically inert material containers for a time not exceeding 6 months.

b) *Solution B2*

The solution shall be prepared by dissolving the indicated amounts of the following salts in water and dilute to a total volume of 7,0 l :

— KCl: 486,2 g (= 69,5 g/l);

— $NaHCO_3$: 140,7 g (= 20,1 g/l);

— KBr: 70,4 g (= 10,0 g/l);

— BO_3H_3: 19,0 g (= 2,7 g/l);

— NaF: 2,1 g (= 0,3 g/l).

The solution shall be stored in well-stoppered glass or other chemically inert material containers for a time not exceeding 6 months.

4.3 Volume ratio

The ratio of the volume of test solution to the total surface area of the test pieces shall be between 3 ml/cm^2 and 6 ml/cm^2.

4.4 Reagent purity

The purity of the gases shall be 99,5 % minimum per volume.

All chemicals shall be of reagent quality.

The water shall be distilled or deionized.

NOTE The conductivity of the water should not normally exceed 5 µS/cm.

5 Apparatus

Testing shall be carried out in apparatus which meets the specific requirements for HIC tests and shall take full account of the safety procedures necessary when using toxic hydrogen sulfide gas.

The basic equipment of the apparatus shall include the following:

— a test vessel and supports made of chemically inert material such as glass or polytetrafluorethylene (PTFE);

— equipment to maintain the standard temperature during the test;

— devices to measure gas flow rates;

— a trap to avoid backstreaming of air which could give oxygen contamination of the test atmosphere. This could be, for instance, a vessel with sodium hydroxide solution to bind the surplus H_2S.

6 Test Pieces

6.1 Location and orientation

6.1.1 Samples from which the test pieces will be machined shall be removed from the material to be assessed by any appropriate method. If the samples are flame cut, they shall be of sufficient size such that test pieces can be machined well away from the heat affected zones.

6.1.2 Three test pieces shall be prepared from the sample (see **2.1**) with the main axis in the longitudinal or in the main deformation direction:

a) for welded tubes test pieces shall be taken from weld and in an angle distance of 90° and 180° to the weld;

b) for seamless tube test pieces shall be circonferentially staggered by 120°;

c) for other product, location and number of test pieces shall be agreed at the time of enquiry and order.

6.1.3 A method shall be used to maintain the identity of the test pieces. This identification shall be made on one or on both small surfaces.

6.2 Size

If not otherwise specified, the dimensions of the test pieces shall be as follows:

— length L = 100 mm ± 2 mm;

— width b = 20 mm ± 0,5 mm;

— thickness a = full material thickness less a maximum of 1 mm on each surface for products with nominal thicknesses from 6 mm up to 30 mm.

The thickness of the test piece shall be at least 50 % of the nominal thickness with a maximum of 30 mm.

For product with thickness lower than 6 mm or greater than 30 mm the dimensions of test pieces shall be defined at the time of enquiry and order.

6.3 Preparation

6.3.1 Mechanical flattening of curved test pieces shall not be permitted. All test pieces shall be premachined to final dimensions plus 0,25 mm on width (b) and thickness (a). The final 0,25 mm shall be removed equally from opposite faces and in stages using a wet wheel surface grinder or equivalent.

6.3.2 The four principal faces of the test pieces shall then be prepared using standard metallographic preparation methods to a final 320 grit finish.

6.3.3 If testing is not to be carried out during the same day the test pieces shall be stored in a dessiccator. It is recommended that storage should not exceed 24 h.

7 Test procedure

7.1 Test batch

When test pieces are tested in batches, the number of test pieces that make up a test batch shall be limited only by the volume of the test vessel and the solution volume/test piece surface area ratio (see **4.3**).

7.2 Degreasing

All test pieces shall be degreased in a suitable solvent immediately prior to testing. Degreasing shall be considerated as satisfactory if the following test is satisfied: A drop of distilled water, which is placed on the test piece surface after degreasing, must spread out, i.e. wet the surface without forming beads.

After degreasing, the test piece shall only be handled using degreased tongs or clean gloves.

7.3 Test piece exposure

7.3.1 The test pieces shall be stacked with the faces which were parallel to the original surface of the product vertical in the test vessel, spaced in accordance with Figure 4 by distancers (made of glass, PTFE or similar material) normally not less than 5 mm apart and 5 mm minimum from the test vessel walls.

7.3.2 The temperature of the test solution shall be maintained at 23 °C ± 2 °C for the period of the test.

7.3.3 Testing shall be carried out using one of the test solutions specified in accordance with clause 4. The test solution to be used shall be agreed at the time of enquiry and order.

7.3.4 Care shall be taken to ensure that all joints of the apparatus are gas tight and hence avoid any possible loss of saturation in the test solution or introduction of oxygen.

7.4 De-aeration and saturation

7.4.1 The solution shall be de-areated by purging with nitrogen at a minimum rate of 100 cm^3/min per litre of solution, for at least 1 h.

7.4.2 After de-aeration H_2S shall be bubbled through the test solution to achieve and maintain saturation. The rate of bubbling shall be at least 200 cm^3/min per litre of solution for 60 min minimum and greater than 5 cm^3/min per litre of solution thereafter.

NOTE The introduction of gases should be in such a way that it is efficient. Smaller flow rate may be admitted if measurements have proved that the saturation is obtained in a similar way and maintained.

7.5 Test period

7.5.1 The test shall be run for 96 h, which will commence after H_2S saturation.

NOTE For different bubbling conditions as defined in **7.4**, the saturation should be checked.

7.5.2 At the end of the test period, the pH value and the H_2S concentration of the test solution shall be measured and recorded. The test shall be deemed to be valid if the following conditions are observed:

— solution A: pH ≤ 4,0;

— solution B: pH = 5,0 to 5,4;

— H_2S concentration ≥ 2 300 ppm for both solutions (measurements shall be by an iodometric method or equivalent).

7.5.3 The test pieces shall be cleaned after finishing the test under running water using hand brushes (plastic) and then dried in air flow. The test piece shall not be pickled.

8 Evaluation of test pieces

8.1 Blisters

The occurence of blisters shall be mentioned in the test report and documented with photographs.

8.2 Test piece sectioning

Transverse sections shall be cut from the test pieces transverse to the main axis at 25 mm, 50 mm and 75 mm positions from a predetermined end, i.e. that with the test piece identity number (Figure 3).

The sections shall be marked to maintain the identification of the test pieces.

8.3 Preparation of sections

Each section shall be polished metallographically through to at least 1 μm diamond finish or equivalent.

Each section shall be cleaned and dried.

NOTE A light etch (2 % nital) may be used.

8.4 Evaluation of cracking

8.4.1 The sections shall be examined for cracking by optical means at an appropriate magnification not exceeding × 100.

8.4.2 For each crack system observed, the extent of longitudinal (E_{LC}) and transverse (E_{TC}) cracking (Figure 1) shall be measured in order to calculate the ratios defined in clause **9**. Any crack of length lower than 0,1 m shall be ignored.

8.4.3 Any cracks associated with surface blisters, which at no point extend more than 1,0 mm from the top or bottom surfaces of a section, shall be ignored.

8.4.4 Any artefacts, such as remaining grinding marks or holes left by inclusions pulled out during metallographic preparation, or other features that cannot be definitely identified as cracks, shall be ignored.

9 Ratio calculations

9.1 The results of the evalutation shall be presented as a series of ratio:

— crack length ratio (CLR) = $\frac{\sum E_{LCi}}{b} \times 100\ \%$;

— crack thickness ratio (CTR) = $\frac{\sum E_{TCi}}{a} \times 100\ \%$;

— crack sensitivity ratio (CSR) = $\frac{\sum (E_{LCi} \times E_{TCi})}{ab} \times 100\ \%$.

9.2 Only stepwise cracks shall be included in the ratio calculations for CTR and CSR.

9.3 The ratios shall be calculated for each section and the result of the evaluation of the test piece shall be the mean value of the three sections.

9.4 The final result of the evaluation shall be the mean of the values of the nine sections.

10 Test report

The following information shall be reported:

a) reference to this European Standard;

b) identification and dimensions of test pieces;

c) test solution;

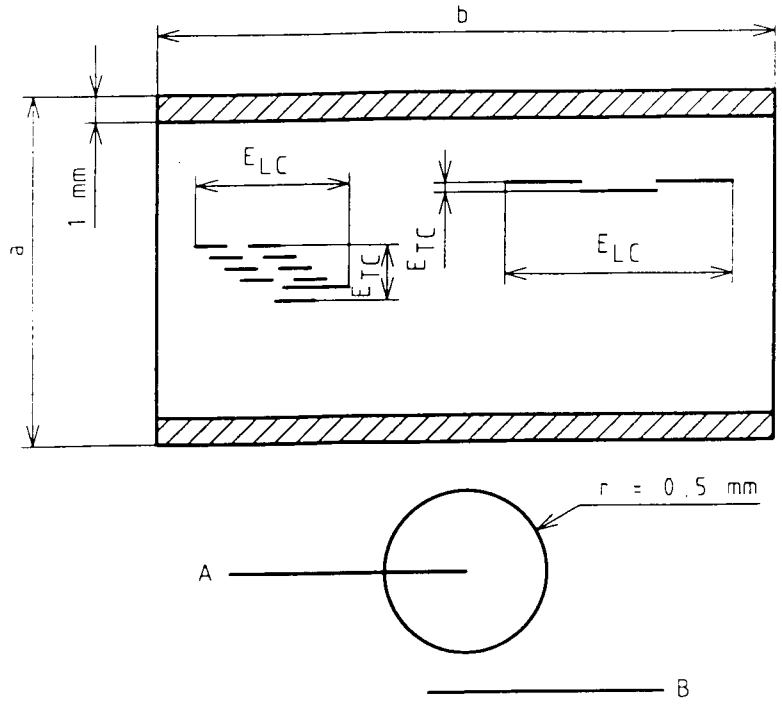
d) pH and H_2S concentration after the test;

e) the final result of the evaluation (see **9.4**);

f) individual values for each section and each test piece with the photographs of the blisters;

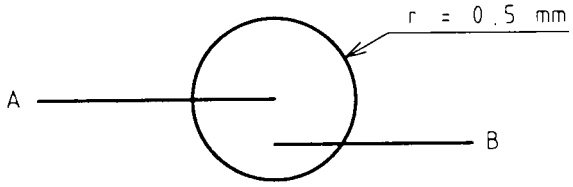
g) whether nital etching was used, mention it (see **8.3**);

h) magnification used for the measurement (see **8.4.1**).



This zone shall be disregarded (see **8.4.2**)

A and B: considered as two separated cracks



A and B: considered as a single crack

Figure 1 — Method of measuring cracks

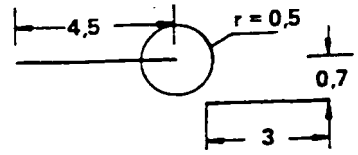
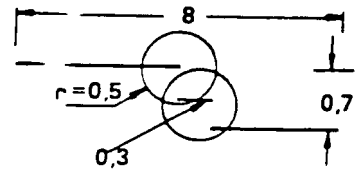
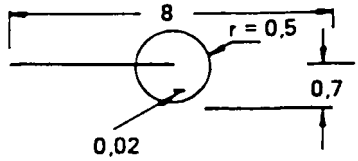
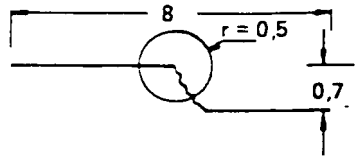
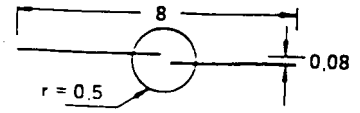
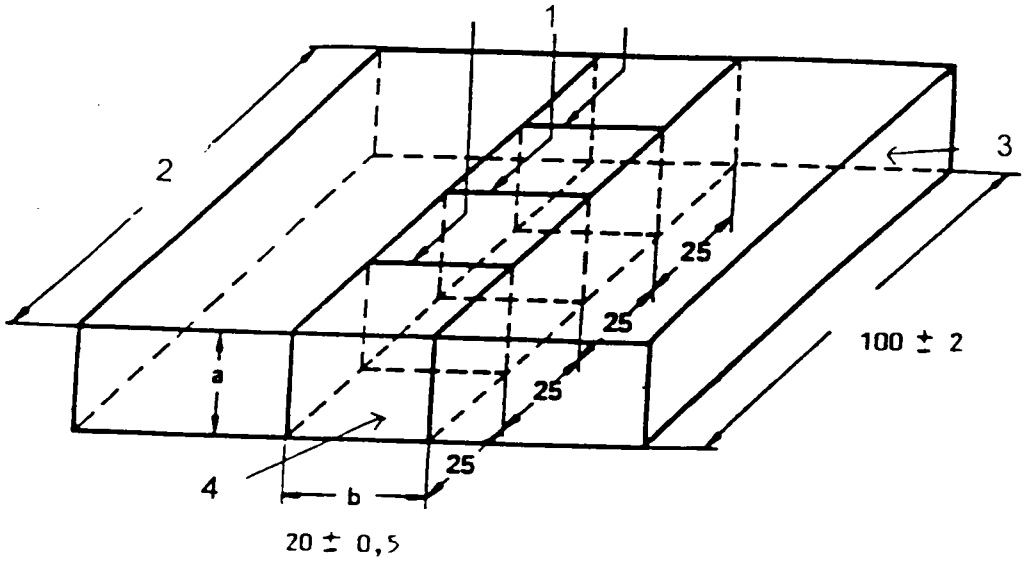
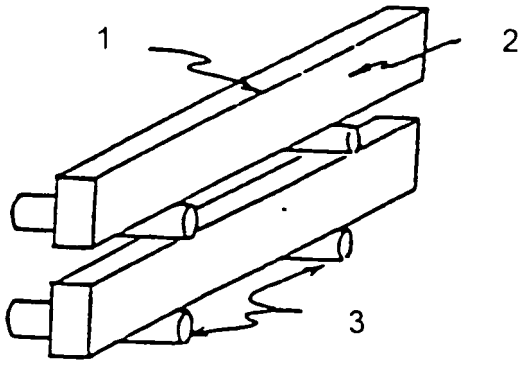
Figure	Discussion [d = distance between two cracks (mm)]	Crack dimensions $E_{LCi} \times E_{TCi}$
 a)	$d > 0{,}5$: two separated cracks	$4{,}5 \times 0/3 \times 0$
 b)	$d < 0{,}5$: crack system	$8 \times 0{,}7$
 c)	the crack of 0,02 mm is less than 0,1 mm so it is not considered	$4{,}5 \times 0/3 \times 0$
 d)	stepwise [as in b)]	$8 \times 0{,}7$
 e)	$d < 0{,}10$: the cracks are considered as a single crack	8×0

Figure 2 — Examples of cracks, illustrating the definitions (see clause **2**)



1) faces to be examined
2) longitudinal or main deformation direction
3) sample
4) section

Figure 3 — HIC test piece dimensions and sectioning procedure



1) face perpendicular to the original surface of the product
2) face parallel to the original surface of the product
3) glass, PTFE or other non-metallic rods

Figure 4 — Arrangement of test pieces in the test solution

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