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Corrosion protection – Carbon and low alloy steels for use in hydrogen sulfide containing environments in oil and gas production – Materials and test methods – Guidelines

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Korrosionsskydd – Kolstål och låglegerade stål för användning i svavelväteinhållande miljöer vid olje- och gasproduktion – Material och provningsmetoder – Vägledning

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E-post: sis.sales@sis.se. Internet: www.sisforlag.se

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English version

**Corrosion protection - Carbon and low alloy steels for use in
hydrogen sulfide containing environments in oil and gas
production - Materials and test methods - Guidelines**

Protection contre la corrosion - Aciers au carbone et peu
alliés pour utilisation en présence de l'hydrogène sulfuré
dans la production de pétrole et de gaz - Matériaux et
méthodes d'essais - Lignes directrices

Korrosionsschutz - Unlegierte und niedrig legierte Stähle
zum Einsatz in H₂S-haltigen Medien in der Öl- und
Gasproduktion - Werkstoffe und Prüfverfahren - Richtlinien

This European Prestandard (ENV) was approved by CEN on 15 September 2000 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 262 "Metallic and other inorganic coatings", the secretariat of which is held by BSI.

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

It is based on EFC 16 "Guidelines on materials requirements for carbon and low alloy steels for H₂S-containing environments in oil and gas production" prepared by the European Federation of Corrosion (EFC) Working Party on Corrosion in Oil and Gas production and published by the Institute of Materials, London, 1995.

It provides guidelines in the form of recommendations. It should not be quoted as if it were a specification and particular care should be taken to ensure that claims of compliance are not misleading.

Over the 3 year period since this project was initiated a number of helpful comments have been received which should improve the clarity of the text. It is intended that these changes will be incorporated in the text should the decision ever be taken to convert this European Prestandard into a European Standard. The changes envisaged are outlined below. Additional changes may be found to be appropriate in response to any further comments that are submitted during the lifetime of the Prestandard.

Reference in prENV	Comments
1 Scope	<p>It is proposed to clarify the <u>role</u> of the tests by deleting the penultimate paragraph and replacing it by:</p> <p>"Procedures for sulfide stress cracking and stepwise cracking testing including acceptance criteria for the various test methods are given for qualifying materials for service. Requirements for quality assurance and quality control testing are outside the scope of this document. Appropriate methods for hardness testing of components and weld zones for service in H₂S containing environments are described."</p>
2 Normative references	<p>References to EN 10003-1 (Metallic materials — Brinell hardness test - Part 1: Test method) and EN 10109-1 (Metallic materials — Hardness test — Part 1: Rockwell test (scales A, B, C, D, E, F, G, H, K) and Rockwell superficial 15 T test (scales 15 N, 30 N, 45 N, 30 T and 45 T)) are to be added.</p> <p>As U-Bends are not covered by this Prestandard references to EN ISO 7539-3 are to be deleted.</p> <p>The Prestandard contains normative references to API 5CT (Specification for casing and tubing), NACE TM0284-96 (Evaluation of pipeline and pressure vessel steels for resistance to hydrogen-induced cracking) and NACE TM0177-96 (Laboratory testing of metals for resistance to environmental cracking in H₂S environments).</p> <p>It is intended that these will be replaced by the following standards: EN ISO 11960 (Petroleum and natural gas industries — Steel pipes for use as casing or tubing for wells); EN 10229 (Evaluation of steel products to hydrogen induced cracking (HIC)); EN ISO 7539 (Corrosion of metals and alloys — Stress corrosion testing), Parts 1 to 4 and 5 to 7.</p>
3.1.28 "stepwise cracking"	<p>The term "stepwise cracking" (SWC) is used throughout this Prestandard. It has been pointed out that NACE has now standardized on the alternative term hydrogen "induced cracking" (HIC). HIC is also the term referred to in EN 10229 although that European Standard does cross reference to this Prestandard and states that the term SWC is used in this document. SWC was the term strongly preferred by the majority of EFC working group members and would still be consistent with EN 10229. Therefore it is proposed to retain the term SWC.</p>

4.2.1 Sulfide stress cracking domains

Second paragraph; insert “in accordance with EN ISO 11960” at the end of the first sentence after “P110”. Figure 1 was first published in 1991(NACE Corrosion '91, Kermani et al) and is based (as stated) on tests on OCTG up to P110 strength.

Figure 1 has been largely accepted as it has also been subsequently validated for some other materials and similar trends in welded materials have been published. The main areas of contention have been a) the horizontal cut off at pH 6,5, and b) the horizontal cut off at pH 3,5 and “traces of H₂S”.

The cut-off at high pH is because there is a change in corrosion behaviour above pH 6,5 and SSC does not arise. High pH values, associated with different cracking mechanisms, are found in refinery operations but these are specifically stated to be outside of the scope of this document.

The cut-off at low pH arises principally because it represents an almost impossible set of conditions, but also because of safety considerations. To reach a pH below 3,5 at pH₂S less than 3,5 mbars requires simultaneously a pCO₂ > 6 bars and a very high condensation rate — conditions which would be rare. Nevertheless, if pH values below 3,5 were achieved then, as stated 4.2.1, paragraph 6, the threshold H₂S level is so low that it could not be practically applied in field conditions. Nevertheless, even if very low levels of H₂S are present, there is a real risk of cracking due to hydrogen entry into the steel.

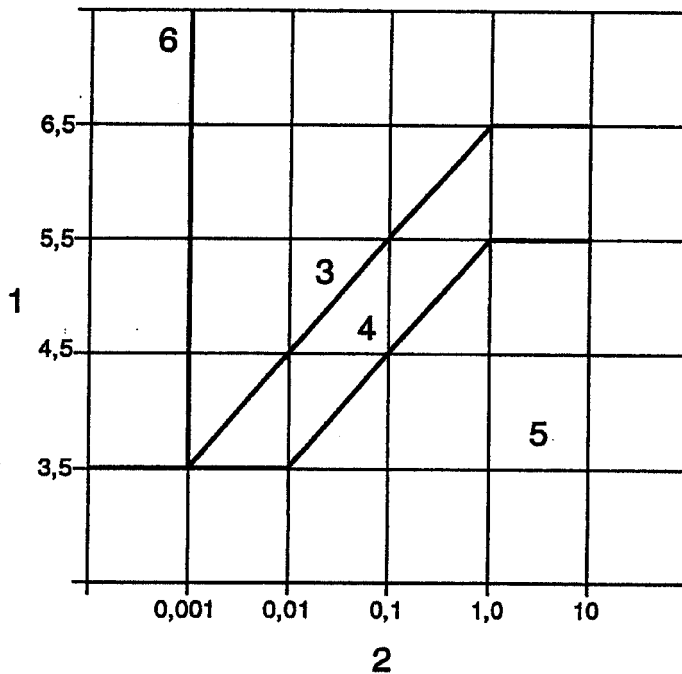
A criticism has been made in the past that the lower limit of the graph was established from subsized diameter test samples (2 mm), which are more sensitive to failure (particularly if they pit or corrode in the test solution), and is therefore too conservative. Proposals have been made to limit the bottom edge of the graph with a vertical line at 0,001 bar H₂S from pH 3,5 downwards but it is considered highly likely that laboratory tests carried out to the requirements stated in the annexes to the Prestandard would result in failures rather than passes on this line and therefore would be inconsistent and unsafe.

The diagram as drawn is fundamentally safe. In defining low pH conditions as potentially sour it draws attention to them. For any application that represents a really substantial investment for which adding sour service requirements would greatly increase the materials cost it then becomes worthwhile to carry out corrosion testing to see if non-sour service grades might actually give acceptable performance. This approach is clearly stated in 4.2.1, paragraph 7, and 5.2.1, paragraph 2. Thus, figure 1 is not ruling the user, it is a guideline. If the user wishes to challenge the figure and the potential cost-saving overall justifies the cost of laboratory tests then 4.2.1 and 5.2.1 allow a final decision to use a specific material to be made on the basis of laboratory test results.

A modified version of the sour service definition graph was published in *Materials Performance* in April 1997. This splits region 3 of figure 1 into a totally “sweet” region where no metallurgical restrictions apply, and a region defined as “slightly sour” with very minor yield strength restriction (YS < 130 ksi). It is suggested that this improved version of the original graph would be adopted, as shown :

4.2.1 (continued)

(Proposed revised) **Figure 1 — Sulfide stress corrosion cracking domains as a function of pH and hydrogen sulfide partial pressure**



NOTE 1 bar = 0,1 MPa

Key

- 1 Solution pH
- 2 Hydrogen sulfide partial pressure (bar)
- 3 Slightly sour service (YS ≤ 130 ksi (895 MPa))
- 4 Moderately sour service (YS ≤ 110 ksi (758 MPa))
- 5 Severely sour service
- 6 Sweet service (no metallurgical restrictions)

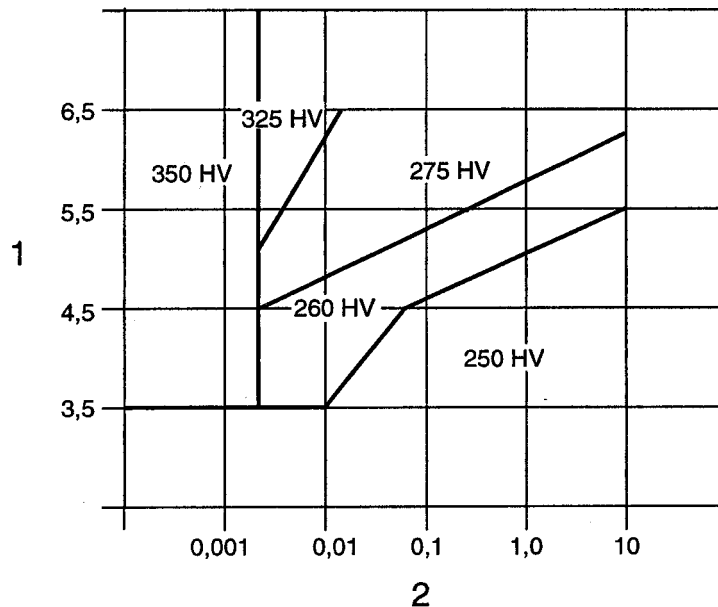
4.2.1 (continued)

It is further proposed to add to the draft a new figure 2 showing hardness limits for different sulfide stress cracking domains for welded items. The text would therefore be modified as follows. Delete 4.2.1, paragraph 7, and substitute the following new paragraphs 7 and 8:

« For welded items the sulfide stress cracking domains are defined by figure 2 with the maximum hardness limits for each of the environmental domains.

For environments or materials that are not adequately covered by figures 1 or 2, the resistance to SSC can be established in accordance with annex B.

(New) **Figure 2 — Hardness limits for different sulfide stress cracking domains for welded items**



Key

- 1 Solution pH
- 2 Hydrogen sulfide partial pressure (bar) »

5.2.2.1 General

The last sentence is ambiguous as it does not say what a “mildly sour” environment is. It is proposed to change the text to “.....hardness values may be relaxed in less sour environments, for example, as indicated in Table 1, or where appropriate testing demonstrates resistance to SSC.”

5.3.2 Seamless pipes, castings and forgings

Add the following new sentence to the end of 5.3.2. “No special limit on sulfur content is required for castings”.

Table B.1 Guidelines on test methods to be used and suggested acceptance criteria

a) Discussion on 90% actual yield

There has been some strong reaction to the acceptance criteria being based on 90 % of the actual yield stress. This point was also debated at length in the early stages of establishing the reference document (EFC 16). The following is a brief summary of the philosophy.

The testing is supposed to be “realistic” and geared to the many fields in Europe that have low levels of H₂S or that are expected to gradually become slightly sour with time. The principal realistic aspect is the control of the test environment in which the partial pressure of H₂S is set at the anticipated field condition, as is the pH of the water phase which is buffered to give stable conditions through the test. By comparison, the established NACE test solution is very acid at the start of the test (pH 2,7) with conditions changing over the 30 days ending at a pH of around 4,5. This can result in excessive and unrealistic hydrogen charging at the start of the test with risk of cracking which might not arise in a less aggressive environment. Where required, the

qualifying test environment can be established at slightly more severe conditions than will arise in practice to provide a margin of safety, but this is the asset owner / material purchaser's responsibility.

Given that the environment is similar to what the material will see in practice then the stress level for testing should also be comparable to real service stress levels. It has been stated that pipelines are designed at 72 % of SMYS and piping codes and pressure vessel codes also design at only fractions of the specified minimum yield stress value, implying that this then represents the stress level in the steel. Actually this simply provides a safety margin for the fact that at local spots the stresses will be much higher. The aim is to ensure that at any point on the design the stress stays below the yield strength of the material. Most specifically, stresses are proven to be high at and around welds. At these locations residual stresses from contraction of the weld metal result in local stress values equal to the actual yield strength of the parent steel. Field pipelines may also be subjected to high thermal stresses which can give rise to movement of the pipe and have been shown to result in plastic deformation of the steel in some cases, obviously exceeding the actual yield strength value of the steel. Similarly the mechanical coupling of OCTG is stressed to the yield point close to the sealing surface.

It is these considerations that resulted in the "90 % of actual yield strength acceptance criteria" being established. Some felt that 100 % actual yield should be set but this would lead to problems in conducting the tests: 90% is considered practical but also consistent with the philosophy of the test approach.

To reassure some commentators on this topic, readers are asked to note the clarifying statement that is to be added to the scope of the Prestandard (see earlier comment relating to "1 Scope"). This points out that the tests are for qualification of materials and not for quality assurance / quality control (QA/QC) testing, which are outside the scope of the document. Thus, fears that these tests will be set for order release purposes should be relieved since that was never intended and would be inappropriate.

b) Discussion on evaluation of welds (third column of table B.1)

FPB and CR samples are not regarded as appropriate for judging fitness for service of non-welded components so it is inconsistent to allow them for welds. Nevertheless, FPB samples are often the only practicable way of evaluating the influence of the weld bead, despite the obvious interference of the geometric effect on the stress field. It is therefore proposed to retain FPB samples (with the specimen transverse to the weld as stated in footnote d) under "Fitness for service", but to put CR samples under the category "Ranking".

B.10.2 Method

In line 1, change "Bent beam specimens" to "Bend specimens".

Annex E

Change "Annex E (informative)" to "Annex E (normative)". In annex E change "should" to "shall" throughout.

General — Normative references

EN ISO 11960

EN ISO 11960 is equivalent to API 5CT. It is currently undergoing revision to bring it up to date with recent changes in API 5CT. There is an intent from both API and ISO that it will be published as a joint ISO/API standard, replacing API 5CT, in the medium future. The second edition of ISO 11960 was issued as a Draft International Standard (DIS) in August 1998.

It is recommended that all references to API 5CT are replaced in the Prestandard with a reference to EN ISO 11960. The grade names are the same in EN ISO 11960 as in API 5CT.

ISO 7539

ISO 7539 is a very broad standard encompassing, apparently, all types of stress corrosion test and test piece. It does not deal with test environments. NACE TM0177 on the other hand deals quite specifically with two test solutions and closely defines the test piece geometry. To use ISO 7539 in place of NACE TM0177 in the Prestandard the following additions will have to be made to the text of the Prestandard.

B.2 Test solutions

At the end of B.2 delete “NACE TM0177” and replace with “table B.1”. Insert new Table B.1 as follows:

(New) **Table B.1 — Solution volume to specimen area ratios and test vessel sizes**

Test method	Solution volume to specimen area ratio (ml/cm ²)	Maximum test vessel capacity (l)
A	20 to 40	—
B	20 to 40	10
C	20 to 40	—
D	10 to 15	10
E	20 to 40	—

B.4 Reagents

Delete “NACE TM0177 and EN ISO 7539-1” and replace with “EN ISO 7539-1 with the following additions:

- a) Solutions shall be de-aerated using an inert gas. The inert gas used shall be nitrogen with a purity of at least 99,998 %.
- b) Test water shall be either distilled or de-ionized water with a purity equal to or greater than the requirement in ASTM D1193 Type IV. (NEED TO REPLACE WITH AN ISO REFERENCE.)
- c) All other gases and chemicals used shall be reagent grade or chemically pure with a minimum purity of 99,5 %.”

B.7 Test vessels

In B.7 delete “NACE TM0177” and replace with “EN ISO 7539-1 and Table B.2.” Add a new paragraph:

“For Methods B and D the H₂S gas shall be introduced into the test solution below the level of the test pieces using a glass diffuser to produce a fine dispersion of gas bubbles.”

B.9 Test Method A

In B.9.1 delete “in accordance with NACE TM0177 (Method A)” and replace with “in general compliance with EN ISO 7539-4 and in accordance with”.

In B.9.4, delete “NACE TM0177” and replace with “annex D and Table 6 of annex D of ISO 6892, annex D, but having a parallel section with a diameter of 6,35 mm over a gauge length distance of 25 mm.”

A comment may be needed on the transition radius since this has been shown to be important.

B.11 Test method D (double cantilever beam testing)**B.11.2 Principle**

EN ISO 7539-6 (Pre-cracked specimens) does not standardize on test piece geometry but provides guidance on valid test piece sizes. "Non-valid" test pieces are permitted if the material is not thick enough. Some further words of guidance on precise test piece geometry may have to be added if the reference to NACE TM0177 is replaced by EN ISO 7539-6.

B.11.3 Fatigue precracking

Delete "NACE TM0177 and".

B.11.4 Specimen loading

EN ISO 7539 advocates the use of constant load tests rather than constant displacement, such as that used in NACE with wedge loading. Reviewers of this Prestandard need to indicate if this is an acceptable change. If so, then B.11.4 can be deleted (as EN ISO 7539-6 is referred to in B.11.2) and the subsequent subclauses renumbered.

B.11.5 Stress intensity factor

Replace "NACE TM0177" with "EN ISO 7539-6"

EN 10229

The view has been expressed that it would not be appropriate to use EN 10229 as a replacement for NACE TM0284 at this time. It is also noted that EN 10229 diverges from NACE TM0284 in a number of ways. Nevertheless, there is a strong desire to apply European Standards when available and so some amendments to annex E of this Prestandard are proposed to account for these differences.

EN 10229, 6.1.2 only makes brief reference to welded pipe: "for welded tubes test pieces shall be taken from weld and in an angle distance of 90° and 180° to the weld" (sic)

In contrast NACE TM0284 fully covers sampling from HFI, SAW and spiral welds in pipe (Section 4) and also describes how to section the test pieces for metallographic examination once the test has been carried out.

It is hoped that such clarifications will be made to the text of EN 10229 in its first revision.

EN 10229, 7.3.2 : The test temperature is (23 ± 2) °C rather than (25 ± 3) °C as in NACE (6.5). This point is already stated in E.3, so E.3 can be deleted (requiring renumbering of subsequent clauses).

EN 10229, 7.5.2 requires that the final pH of the test solution after the test is pH 5,0 to 5,4 rather than the pH 4,8 to 5,4 required by NACE (6.3). This seems to be an unnecessary change, but it is acceptable.

EN 10229, 8.2 : Test piece sectioning after test makes no allowance for seam welds in pipe. NACE fully describes how to section through welds for micro-examination (Section 4, figures 3, 4 and 6). EN 10229 leaves these decisions to the manufacturer / test laboratory.

It is hoped that such clarifications will be made to the text of EN 10229 in its first revision.

EN 10229, 8.4.2 : Any crack <0,1 mm long is to be ignored. NACE requires all identified cracks to be included in the calculations for cracking susceptibility (7.3). A steel producing only very short cracks during the HIC test still has a questionable resistance to hydrogen induced cracking.

This Prestandard already places emphasis on identification of all significant cracks (E.6, paragraph 2). It is proposed to add a final sentence in this section stating, "All identified cracks are to be included in the calculations for cracking susceptibility".