



Designation: A1084 – 13

Standard Test Method for Detecting Detrimental Phases in Lean Duplex Austenitic/ Ferritic Stainless Steels¹

This standard is issued under the fixed designation A1084; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The purpose of this test method is to allow detection of the presence of detrimental chromium-containing phases in selected lean duplex stainless steels to the extent that toughness or corrosion resistance is affected significantly. Such phases can form during manufacture and fabrication of lean duplex products. This test method does not necessarily detect losses of toughness nor corrosion resistance attributable to other causes, nor will it identify the exact type of detrimental phases that caused any loss of toughness or corrosion resistance. The test result is a simple pass/fail statement.

1.2 Lean duplex (austenitic-ferritic) stainless steels are typically duplex stainless steels composed of 30-70 % ferrite content with a typical alloy composition having Cr > 17 % and Mo < 1 % and with additions of Nickel, Manganese, Nitrogen and controlled low carbon content as well as other alloying elements. This standard test method applies only to those alloys listed in [Table 1](#). Similar test methods for some higher alloyed duplex stainless steels are described in [ASTM A923](#), but the procedures described in this standard differ significantly for all three methods from the ones described in [A923](#).

1.3 Lean duplex stainless steels are susceptible to the formation of detrimental chromium-containing compounds such as nitrides and carbides and other undesirable phases. Typically this occurs during exposures in the temperature range from approximately 300 to 955°C (570 to 1750°F) with a maximum susceptibility in the temperature range around 650 to 750°C (1200 to 1385°F). The speed of these precipitation reactions is a function of composition and the thermal or thermo-mechanical history of each individual piece. The presence of an amount of these phases can be detrimental to toughness and corrosion resistance.

1.4 Because of the low molybdenum content, lean duplex stainless steels only exhibit a minor susceptibility to sigma or other types of molybdenum containing intermetallic phases. Heat treatment, that could lead to formation of small amounts

of molybdenum containing intermetallics, would result in a large amount of precipitation of detrimental nitrides or carbides – long before any signs of sigma and similar phases would be observed.

1.5 Correct heat treatment of lean duplex stainless steels can eliminate or reduce the amount and alter the characteristics of these detrimental phases as well as minimizing Cr-depletion in the matrix phase in the immediate vicinity of these phases. Adequately rapid cooling of the product from a suitable annealing temperature provides the maximum resistance to formation of detrimental phases by subsequent thermal exposures. For details of the proper annealing temperature recommendations for the alloy and product in question, the user is referred to the relevant applicable ASTM product specification.

1.6 Compliance with the chemical and mechanical requirements for the applicable product specification does not necessarily indicate the absence of detrimental phases in the product.

1.7 These test methods include the following:

1.7.1 *Test Method A*—Etch Method for detecting the presence of potentially detrimental phases in Lean Duplex Stainless Steels

1.7.2 *Test Method B*—Charpy V-notch Impact Test for determining the presence of detrimental phases in Lean Duplex Stainless Steels.

1.7.3 *Test Method C*—Inhibited Ferric Chloride Corrosion Test for determining the presence of detrimental phases in Lean Duplex Stainless Steels.

1.7.4 Examples of the correlation of thermal exposures, the occurrence of detrimental phases, and the degradation of toughness and corrosion resistance are given in [Appendix X2](#), [Appendix X3](#) and the References.

1.8 Guidelines for the required data needed for subcommittee A01.14 to consider listing a lean duplex stainless steel in this standard test method are given in [Annex A1](#).

1.9 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to other units that are provided for information only and are not considered standard.

1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

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TABLE 1 List of the Lean Duplex Grades Covered by this Standard

Grades
UNS S32101, UNS S32304

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

A923 Test Methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic/Ferritic Stainless Steels

E23 Test Methods for Notched Bar Impact Testing of Metallic Materials

G48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution

3. Terminology

3.1 Definitions:

3.1.1 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology G15 and Terminology E6. Definitions provided herein and not given in Terminology G15 or in E6 are limited only to this standard.

4. Significance and Use

4.1 Test Method A shall only be used to supplement the results of Test Methods B and C. It shall not be used as a rejection criterion, nor shall it be used as an acceptance criterion. Test Methods B and C are intended to be the procedures giving the acceptance criteria for this standard.

4.2 Test Method A can reveal potentially detrimental phases in the metallographic structure. As the precipitated detrimental phases can be very small, this test demands high proficiency from the metallographer, especially for thinner material.

4.3 The presence of detrimental phases is readily detected by Test Methods B and C provided that a sample of appropriate location and orientation is selected.

4.4 The tests do not determine the precise nature of the detrimental phase but rather the presence or absence to the extent that the normally expected toughness and corrosion resistance of the material are significantly affected.

4.5 This standard covers testing of samples taken from coil, coil- and plate mill plate, sheet, tubing, piping, bar and deformed bar, though some of these products might not be suitable for testing according to Method B (see Test Method B for further details). Other product forms have thus far not been sufficiently tested and documented to be an integral part of this standard, though the standard does not prohibit testing of these

product forms according to the three test methods. For these other product forms, this standard gives only limited and non-exhaustive guidance as to interpretation of result and associated acceptance criteria.

4.6 Testing on product forms outside the present scope of this standard shall be agreed between purchaser and supplier.

5. Sampling, Test Specimens, and Test Units

5.1 Sampling:

5.1.1 Because the occurrence of detrimental phases is a function of temperature and cooling rate, it is essential that the tests be applied to the region of the material experiencing the conditions most likely to promote the formation of detrimental phases. In the case of common heat treatment, this region can be that which cooled most slowly or undergoes extremely rapid cooling.

5.1.2 For practical purposes, it is considered sufficient that the sampling location for flat mill products be from a location that is at least twice the material thickness from the as-heated edges.

5.1.3 Purchaser and supplier may agree on more detailed rules regarding the sampling location.

5.1.4 The number of samples as well as frequency of sampling shall be agreed between purchaser and supplier of the material.

5.2 Test Specimens and Test Units:

5.2.1 Details of test specimen and test unit requirements are listed together with each of the Test Methods A, B and C.

TEST METHOD A—ETCH METHOD FOR EVALUATION OF THE PRESENCE OF POTENTIALLY DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

6. Introduction

6.1 The etch test in this standard shall only be used for exploratory purposes. The reason for this is the small size of the detrimental phases typically occurring in lean duplex stainless steels and the difficulty in achieving a fully reproducible etch structure, which depends on factors such as specimen size and geometry, etching current and potential, composition of the lean duplex as well as the amount and type of detrimental phases present. The test method contained in this standard is, however, the best known metallographic procedure to show the appearance and approximate amount of detrimental phases in a lean duplex stainless steel.

6.2 As there is no formal test result from the metallographic etch method, the actual test method is attached to this standard as **Appendix X1**.

TEST METHOD B—CHARPY V-NOTCH IMPACT TEST FOR DETERMINATION OF THE PRESENCE OF DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

7. Scope

7.1 This test method describes the procedure for conducting the Charpy V-notch impact test as a method of detecting the

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

precipitation of detrimental phases in lean duplex stainless steels. The presence or absence of an indication of a detrimental phase in this test is not necessarily a measure of performance of the material in service with regard to any property other than that measured directly. The Charpy V-notch procedure as applied here is different from that commonly applied for the determination of toughness and shall not be used when characterization of material toughness is the purpose of the testing.

8. Significance and Use (Test Method B)

8.1 The Charpy V-notch impact test may be used to evaluate mill products, provided that it is possible to obtain a specimen of the proper size from a relevant location.

8.2 Charpy V-notch impact toughness of a material is affected by factors other than the presence and absence of detrimental phases. These factors are known to include different compositions, even when the material is in fully annealed condition; small and otherwise acceptable variations in austenite/ferrite balance; and the lamellar distance between phases. Testing transverse and longitudinal test specimens from mill products can also give different absolute levels of impact toughness.

8.3 **Table 2** indicates the applicability and acceptance criteria for Test Method B. These acceptance criteria have been shown to allow for the natural variation of impact toughness in sound material tested in the transverse direction on plate and in the longitudinal direction on bar and deformed bar, while still being able to identify whether detrimental amounts of undesirable phases are present.

8.4 Acceptance criteria for Test Method B for other products including mill welded pipe, weldments and weld metal are not presently covered by this standard, though purchaser and supplier may agree upon an acceptance criteria (see **Note 1**). Note that the results of weldment testing will depend on the filler metal or weld deposit chemistry.

8.5 Acceptance criteria of sub-size specimens are not covered by this standard, though purchaser and supplier may agree upon a proper conversion factor of the given acceptance criteria in **Table 2**. Conversion factors generally vary by product type and dimensions of product for which the sub-size specimen sampling is needed (see **Note 2**).

NOTE 1—As no data has been presented to A01.14 for welded mill products or other products, no recommendation can be given as to the acceptance criteria for these products. Any acceptance criteria and other details of the test should be supported with data from a pre-qualification

test in line with the minimum requirements of **Annex A1** in this standard.

NOTE 2—As stated in **A370**, Appendix A5.3.3 and ASTM **E23**, Appendix X1.3, there is no general correlation between impact values obtained with specimens of different size or shape. However, limited correlations may be established for specification purposes on the basis of special studies of particular materials and particular specimens. It is commonly seen that the conversion factor is set directly proportional to the ratio between standard and sub-size specimen fracture surface area or a percentage thereof, though whether this is an acceptable way forward to still be able to identify the presence or absence of detrimental phases needs to be documented.

9. Apparatus

9.1 The test apparatus shall be as described in Test Methods and Definitions **A370**.

10. Test Specimens

10.1 *General requirements (all products):*

10.1.1 The test specimen shall be as described in Test Methods and Definitions **A370**.

10.1.2 An impact test for the purpose of detecting detrimental phases shall consist of a single specimen taken from the product piece or lot to be represented.

10.1.3 Provided purchaser and supplier have agreed upon a proper acceptance criterion, sub-size specimens may be used for products with thickness less than that of full-size Charpy V-notch specimen. Required energy for sub-size specimens shall be established and agreed upon based on the specific product type and geometry in question.

10.2 *Flat products (sheet, coil, plate):*

10.2.1 The specimen shall be prepared in the transverse direction. The notch shall be perpendicular to the major rolled surface.

10.3 *Non-deformed bar products:*

10.3.1 The specimen shall be prepared in the longitudinal direction.

10.4 *Deformed bar products:*

10.4.1 The specimen shall be prepared in the longitudinal direction.

10.5 *Other products including mill pipe:*

10.5.1 When this test is applied to a welded structure or to any product having a less than uniform structure, particular attention shall be paid to the location of the V-notch. For example, in the heat-affected zone of a weld, the degree of detrimental phase formation can vary significantly over short distances as a function of the local thermal cycle. In such cases, the placement of the V-notch can affect the measured result significantly.

10.5.2 Following the guidelines of **A370**, the specimen preparation method shall be agreed between purchaser and supplier.

11. Procedure

11.1 Perform the test for a single specimen in accordance with the procedures described in ASTM **A370**, Standard Test Methods and Definitions for Mechanical Testing of Steel Products.

11.2 The test temperature shall be as specified in **Table 2** or lower (see also section **12.3**) for the grade being evaluated.

TABLE 2 Applicability and Acceptance Criteria for Test Method B

Grade	Sampling Location	Test Temperature	Minimum Impact Energy ^A
S32101	base metal	Room temperature ^B	70 J (50 ft-lb)
S32304	base metal	Room temperature ^B	100 J (75 ft-lb)

^A Energy for a full-size specimen tested in transverse direction for flat rolled products and tested in the longitudinal direction for bar products. Required energy for a sub-size specimen is discussed further in section **10.1.3** and **Note 2**.

^B In this standard, room temperature is defined as the temperature range 23 ± 5°C (73 ± 9°F).

12. Acceptance Values and Retests

12.1 Unless otherwise specified, the acceptance criteria shall be as given in **Table 2**.

12.2 If a test specimen shows a value below the specified minimum, one retest of two specimens is permitted. For acceptance, both retest specimens shall show a value at or above the specified minimum value.

12.3 Testing at lower temperatures than indicated in **Table 2** is permissible and if the obtained impact energy is higher than the acceptance criteria indicated in **Table 2** then the sample is approved in accordance with Test Method B of this test method. Failure at a lower test temperature than indicated in **Table 2** does not imply that the sample has failed Test Method B, but only that the test shall not be counted as a proper test and a new test shall be performed at the temperature specified in **Table 2**.

12.4 A product that has failed the Charpy V-notch impact test may be given a full anneal and retested at the option of the supplier.

TEST METHOD C—INHIBITED FERRIC CHLORIDE CORROSION TEST FOR DETERMINATION OF THE PRESENCE OF DETRIMENTAL PHASES IN LEAN DUPLEX STAINLESS STEELS

13. Scope

13.1 This test method describes the procedure for conducting an inhibited ferric chloride corrosion test for detecting the presence of detrimental phases in lean duplex stainless steels. The presence or absence of corrosion attack in this test is not necessarily a measure of performance of the material in other corrosive environments; in particular, it does not provide a basis for predicting resistance to forms of corrosion not associated with the precipitation of detrimental phases (see **Note 3**).

13.2 The test method uses a ferric chloride solution inhibited by addition of sodium nitrate, since a standard ferric chloride solution is too aggressive to give valuable results for lean duplex stainless steels.

NOTE 3—Although this test method uses some equipment and procedures similar to those of Test Methods **G48**, this test method shall not be confused with Test Methods **G48**. This test method does not determine the critical pitting temperature or test for the suitability for use in a particular environment. This test method is designed solely for detection of the precipitation of detrimental phases in lean duplex stainless steels. The inhibited solution might not give the same ranking as the uninhibited solution, when comparing a range of stainless steels, but it can reveal the presence of deleterious phases in the lean duplex stainless steels listed in **Table 1**.

14. Significance and Use (Test Method C)

14.1 The inhibited ferric chloride corrosion test may be used to evaluate mill products as well as fabricated products, provided that it is possible to obtain a specimen from a relevant location and having the proper geometry.

14.2 **Table 3** indicates the applicability and acceptance criteria for Test Method C.

TABLE 3 Applicability and Acceptance Criteria for Test Method C

Grade	Sample Location	Test Temperature	Maximum Acceptable Corrosion Rate Calculated from Weight Loss
S32101	base metal	25°C (77°F)	10 mdd ^A
S32304	base metal	25°C (77°F)	10 mdd ^A

^A For a definition of "mdd" – see **Note 4**.

14.3 Acceptance criteria for weldment and weld metal shall be agreed upon between purchaser and supplier of the product in question prior to testing. Results obtained from testing of weldments also depend on the filler metal and weld deposit chemistry.

15. Apparatus

15.1 *Glass Beakers*, 1000 mL, tall-form, or Erlenmeyer flasks, 1000 mL, wide neck, or 50-mm (2-in.) diameter test tubes, or other suitable glass containers.

15.2 *Glass Cradles* (**Fig. 1**)—The dimensions of the cradle shall be restricted to those that permit its passage through the test container opening, a diameter of approximately 40 mm (1.6 in.) in the case of the Erlenmeyer flask.

15.3 *Constant Temperature Device*—Water or Oil bath or other device that ensures constant temperature of solution and specimen.

16. Inhibited Ferric Chloride Test Solution

16.1 Dissolve 55.1 g of reagent-grade ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, and 6.6 g of reagent-grade sodium nitrate, NaNO_3 , in 600 mL of distilled water (approximately 5% FeCl_3 and 1% NaNO_3 by weight). Filter the solution through glass wool or filter paper to remove insoluble particles.

17. Test Specimen

17.1 *General requirements (all products):*

17.1.1 Various shapes and sizes of test specimens may be used.

17.1.2 After the specimens are cut, any material that might have been affected by high temperature or deformation associated with the cutting shall be removed by machining or polishing prior to testing. This procedure shall include rounding of sharp edges with care taken to remove all burrs.

17.1.3 For all polishing procedures, wet polishing is preferred. If used, dry polishing shall be performed slowly to prevent overheating.

17.1.4 All polishing shall be done to a 120-grit finish or finer.

17.1.5 For other than mill products, testing of a specimen with the surface in the as-fabricated condition can be relevant. However, if oxide scales from heat treatment or weldments are visibly present, then they shall be removed by polishing prior to testing as the oxide scale is likely give rise to a weight loss higher than 10 mdd despite the fact that no actual attack on the metallic part of the specimen is occurring.

17.2 *Flat products (coil, plate, sheet):*

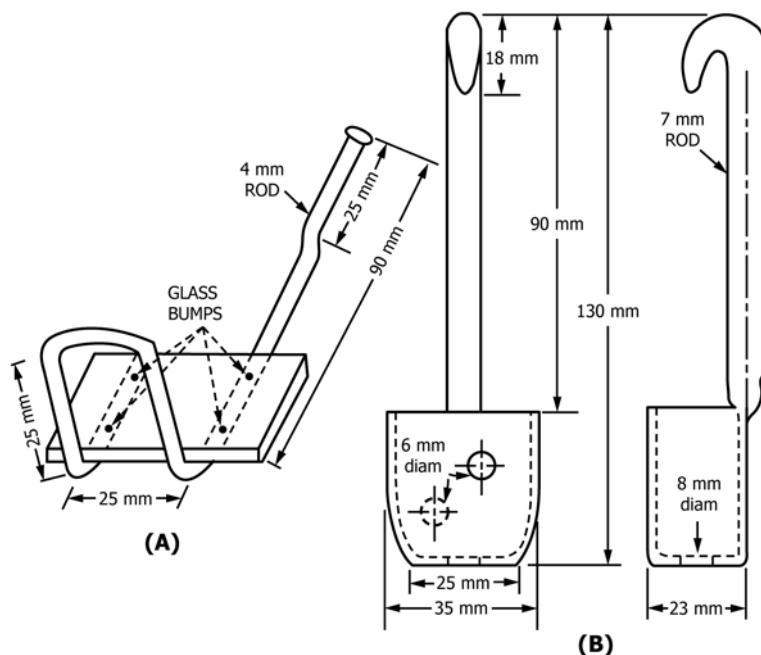


FIG. 1 Examples of Glass Cradles That Can Be Used to Support the Specimen

17.2.1 The test specimen shall be approximately 25 by 50 mm (1 by 2 in.) by thickness. The full thickness of the product shall be included if practical. In the case of thicker sections, the specimen shall be taken in a perpendicular orientation so that the thickness of the product becomes the longest dimension of the specimen resulting in approximate specimen dimensions of 6 mm by 25 mm (1/4 by 1 in.) by product thickness. In very thick sections, the thickness dimension of the specimen shall be cut so that one-half to two-thirds of the product thickness is tested resulting in approximate specimen dimensions of 6 mm by 25 mm (1/4 by 1 in.) by one-half to two-thirds product thickness.

17.2.2 All surfaces shall be polished to a uniform finish.

17.3 *Non-deformed bar products:*

17.3.1 All surfaces of the specimen shall be polished to a uniform finish.

17.4 *Mill tube and pipe products:*

17.4.1 The ID side shall be left as is, whereas cut and OD surfaces shall be polished to a uniform finish.

17.5 *Mill deformed bar products:*

17.5.1 For practical reasons, the default specimen preparation shall be to let the deformed surface be as is, whereas cut surfaces shall be polished to a uniform finish.

17.5.2 If agreed between purchaser and seller, the deformed surface may be machined and polished to a 120-grit finish, or finer.

17.6 *Other product forms:*

17.6.1 Test specimens shall be cut into test specimens convenient for testing, provided that the specimen exposes surfaces representative of the full thickness of the product.

17.6.2 All surfaces of the specimen shall be polished to a uniform finish.

17.7 *Test specimen preparation – after cutting and polishing:*

17.7.1 Subsequent to polishing of the required surfaces, the surfaces of the specimen shall not be chemically passivated by any treatments such as nitric, citric, or phosphoric acid, or pickled by treatments such as nitric/hydrofluoric acid mixture or other pickling acids.

17.7.2 Measure the dimensions of the specimen and calculate the total exposed surface area.

17.7.3 Clean the specimen with magnesium oxide paste or equivalent. Rinse the specimen well with water followed by a dip in alcohol or acetone. Finally dry the specimen in air. The specimen shall be weighed to a precision of 0.001 g or better. Store the specimen in a desiccator until ready for testing.

18. Procedure

18.1 Perform the test using the nitrate-inhibited ferric-chloride solution with a volume at least the larger of 150 mL or 20 mL/cm² (125 mL/in.²) of the specimen surface area. Fill the test container with the required volume of solution, transfer to the constant temperature bath, and allow it to come to equilibrium at the desired test temperature.

18.2 Unless otherwise specified, test temperatures shall be used as indicated for each type of steel in Table 3, maintained within an accuracy of ±1°C (2°F) during the test.

18.3 Place the specimen in the glass cradle and immerse in the test solution once the temperature has been established. Maintain the test temperature throughout the test. Cover the test container with a watch glass during the test period. Unless otherwise specified, the test period shall be 24 h.

18.4 At the end of the 24-h test period, remove the specimen from the solution, rinse with water, scrub with a soft bristle

brush under running water to remove corrosion products, dip in acetone or alcohol, and dry in air. Ultrasonic cleaning is a permitted alternative when there are corrosion products that are difficult to remove.

18.5 Weigh the specimen to a precision of 0.001 g or better and reserve for examination.

19. Acceptance Values

19.1 The corrosion rate is calculated in accordance with the weight loss and total surface area (see [Note 4](#)). The calculated corrosion rate shall not exceed the indicated maximum value (see [Note 5](#)) for the type of stainless steel in question as indicated in [Table 3](#).

NOTE 4—The corrosion rate is calculated in accordance with the following:

$$\text{corrosion rate (mdd)} = \text{weight loss (mg)} / [\text{specimen area (dm}^2\text{)} \times \text{time (days)}]$$

NOTE 5—It is probable that corrosion occurs in the form of pitting. The calculation of a uniform corrosion rate is an inappropriate method of expressing the rate of pitting corrosion. However, in this case, the calculation of a corrosion rate is used primarily to normalize the weight loss for the variety of specimen sizes and shapes permitted.

19.2 If the specimen shows a corrosion rate in excess of the maximum allowed valued as found in [Table 3](#), one retest on two new specimens from the same product is permitted. No retest specimen shall exhibit a corrosion rate in excess of the maximum allowed value.

19.3 Testing temperatures higher than indicated in [Table 3](#) are permissible. If such a test has a corrosion rate less than that specified in [Table 3](#), then the sample is approved in accordance with Test Method C of this specification. Failure at a test temperature higher than indicated in [Table 3](#) does not imply that the sample has failed test Method C, but only that the test shall not be counted as a proper test and shall be redone at the temperature specified in [Table 3](#). The same test specimen shall not be reused for testing at a lower temperature.

19.4 At the option of the producer, a product that has failed the inhibited ferric-chloride corrosion test may be given a full anneal and retested as if it had not been tested before.

19.5 When testing materials in the as-fabricated condition, weight loss can occur with no visible pitting. The cause can be surface inhomogeneities or contamination. In such cases a retest shall be performed after removing the entire original surface and finishing the surface by grinding to a 120-grit finish.

20. Report for Test Methods A, B and C

20.1 *Test Method A*—No test result is reported for this test method. But the specimen size, the etching parameters used and the magnification used should be recorded together with at least one representative image of the etched microstructure for documentation.

20.2 *Test Method B*—Report the test result as being either passed or failed based on the recorded impact toughness relative to the specified acceptance criteria. For informational purposes, record the test temperature and the measured impact toughness.

20.3 *Test Method C*—Report the test result as being either passed or failed based on the recorded corrosion rate relative to the specified acceptance criteria. For failed specimens, record the general position (what surface, edge, or both) of the corroded area; otherwise, report the failed specimen having a “non-visible” attack. For informational purposes, record the test temperature, duration of the test, the measured weight loss and the calculated corrosion rate in “mdd.”

21. Precision and Bias for Test Method A, B and C

21.1 *Precision and Bias*—No information is presented about either the precision or bias of Standard Test Method A1084 for the microstructure evaluation, nor for the pass/fail results of impact toughness and corrosion testing in Test Methods A, B and C since the test results are non-quantitative.

22. Keywords

22.1 acceptance testings; detrimental phases; duplex stainless steels; etch methods; ferric chloride testings; impact toughness testings; inhibited ferric chlorides; lean duplexes; microstructure evaluations; secondary phases; stainless steels

ANNEX

(Mandatory Information)

A1. GUIDELINES REGARDING REQUIREMENTS FOR INCORPORATING NEW ALLOYS AND PRODUCTS

A1.1 When introducing new alloys or products to be incorporated into this standard the following minimum requirements shall be considered by the responsible subcommittee.

A1.2 Any new alloy submitted for incorporation shall be consistent with the overall description of the term “lean duplex alloy” as described in section 1.2 of this standard and shall be identified specifically with its UNS number in [Table 1](#).

A1.3 Appropriate maximum or minimum test temperatures and associated acceptance criteria shall be supplied for both Test Methods B and C respectively together with information on the test specimen preparation used.

A1.4 Examples of microstructural evaluation shall be presented to the subcommittee to validate that the existing examples of microstructure shown in [Appendix X2](#) are relevant