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Petroleum, petrochemical and natural gas industries — Qualification testing and acceptance criteria for protective coating systems under insulation

Industries du pétrole, de la pétrochimie et du gaz naturel — Essais de qualification des systèmes de revêtement protecteurs sous isolation des systèmes de revêtement protecteurs de la protecteur de la protecteu



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents)

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.



Introduction

Unprotected carbon steel in insulated service with the presence of water and concentrating contaminants from the atmosphere or surrounding sources can cause accelerated corrosion and lead to severe metal loss. Additionally, unprotected austenitic and duplex stainless steels can suffer external chloride-induced stress corrosion cracking if contaminates, such as chlorides from the atmosphere and or the insulation, are present at the steel surface. Therefore, steel structures under insulation are normally protected to prevent corrosion-related damage during the operational life required of the equipment.

There are different ways of protecting steel structures from corrosion under insulation. This document deals with protection by use of coating when used as part of a system, including insulation and cladding materials, which can work together to prevent corrosion under insulation (CUI). All components of the corrosion prevention system are important in achieving adequate corrosion protection. This document only deals with the coating part of the corrosion protection system with focus on typical CUI coating environments. Further, this document focuses on accelerated testing protocols and acceptance criteria, so that interested parties can make informed decisions.

In order to ensure effective corrosion protection of steel structures and equipment, it is necessary for owners of such structures, planners, consultants, companies carrying out corrosion protection work, inspectors of protective coatings and manufacturers of coating materials to have at their disposal state-of-the-art information in a concise form on corrosion protection by coating systems. Such information has to be as complete as possible, unambiguous and easily understandable to avoid difficulties and misunderstandings between the interested parties with the practical implementation of protection work.

This document is intended to give the abovementioned information to people who have some technical knowledge of coatings and the process operations of the equipment. It is assumed that the user of this document is familiar with other relevant International Standards, in particular those dealing with surface preparation, inspection/testing of coatings, and relevant regulations.

Future parts of this document are planned to be developed and can include other subjects like higher temperature, cyclic and intermittent services testing of coatings for maintenance and repair, tape-applied coating materials, etc.

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Petroleum, petrochemical and natural gas industries — Qualification testing and acceptance criteria for protective coating systems under insulation

1 Scope

This document describes various corrosion under insulation (CUI) environments in refineries and other related industries and environments, and establishes CUI environmental categories including operating temperature ranges from -45 °C to 204 °C for topside and aboveground service only. This document specifies both established and other test methods for the assessment of coatings used for prevention of CUI for each given environment. This document also provides acceptance criteria for each CUI environment.

NOTE The test results and acceptance criteria can be considered an aid to the selection of suitable coating systems. For service or peak temperatures below –45 °C an optional cryogenic test can be incorporated and for over 204 °C testing acceptance criteria can be agreed between interested parties. Additional or other test and acceptance measures are possible, but require particular agreement between the interested parties.

This document covers spray-applied coatings applied on new carbon and austenitic stainless steel for use in CUI service. This document does not cover testing of sacrificial coatings, such as inorganic zinc, as these coatings can be consumed quickly in wet environments. Developing accelerated corrosion testing for what can be continuous wet service with sacrificial coatings is beyond the scope of this document.

"Non-through porosity" thermal spray aluminium coatings with greater than 250 µm dry film thickness can be tested and qualified in accordance with this document. This document does not cover tape and sheet applied products for use in preventing CUI.

This document does not deal with other aspects of coating degradation, such as those caused by abrasion, erosion, ultraviolet degradation or other methods that can exist given specific environment and construction methods.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 554, Standard atmospheres for conditioning and/or testing — Specifications

ISO 1513, Coatings and varnishes — Examination and preparation of samples for testing

ISO 2409, Coatings and varnishes — Cross-cut test

ISO 2812-2, Coatings and varnishes — Determination of resistance to liquids — Part 2: Water immersion method

ISO 4624, Coatings and varnishes — Pull-off test for adhesion

ISO 4628-2, Coatings and varnishes — Evaluation of degradation of coating coatings — Designation of intensity, quantity and size of common types of defect — Part 2: Designation of degree of blistering

ISO 4628-3, Coatings and varnishes — Evaluation of degradation of coating coatings — Designation of intensity, quantity and size of common types of defect — Part 3: Designation of degree of rusting

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ISO 4628-4, Coatings and varnishes — Evaluation of degradation of coating coatings — Designation of intensity, quantity and size of common types of defect — Part 4: Designation of degree of cracking

ISO 4628-5, Coatings and varnishes — Evaluation of degradation of coating coatings — Designation of intensity, quantity and size of common types of defect — Part 5: Designation of degree of flaking

ISO 4628-8, Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 8: Assessment of degree of delamination and corrosion around a scribe

ISO 7384, Corrosion tests in artificial atmospheres — General requirements

ISO 9227, Corrosion tests in artificial atmospheres — Salt spray tests

ISO 12944-6, Paints and varnishes — Corrosion protection of steel structures by protective paints stems — Part 6: Laboratory performance test methods and associated assessment criteria

ISO 15528, Paints, varnishes and raw materials for paints and varnishes — Sampling

ISO 19840, Paints and varnishes — Corrosion protection of steel structures by protective paint systems — Measurements of, and acceptance criteria for, the thickness of dry films on rough surfaces

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

artificial ageing

procedure designed to accelerate the ageing of a coating system, i.e. to reduce the corrosion-protective efficiency more rapidly than by natural weathering

[SOURCE: ISO 12944-6:1998, 3.1, modified — 'paint system' has been changed to 'coating system'.]

3.2

corrosion under insulation

CUI

corrosion that is a result of the effect of moisture and contaminants, on the steel surfaces under thermal insulation

3.3

dry film thickness

DFT

thickness of a coating remaining on the surface when the coating has hardened

3.4

durability

expected life of a protective coating system to the first major maintenance coating

[SOURCE: ISO 12944-8:2017, 3.3, modified — 'paint system' has been changed to 'coating system' and 'maintenance painting' has been changed to 'maintenance coating'.]

3.5

nominal dry film thickness

NDFT

dry film thickness specified for each coat or for the whole coating system

3.6

peak temperature

maximum temperature for the designed system, including possible upsets and temperature reached as a result of maintenance efforts such as steam cleaning

3.7

sacrificial coating

coating that provides corrosion protection by sacrificing or being consumed in the act of protecting the substrate

4 Performance testing design

4.1 Relationship between artificial testing and natural exposure

The selection of a coating system for specific conditions should preferably be based on experience from the use of the system in similar cases. The reason is that the durability of a CW coating system depends on many external factors, such as the environment, the design of the structure, the insulation material, the weather proofing (cladding), the surface preparation, the application, drying procedures, service temperature, thermal shock, thermal cycling, peak temperature, amount of moisture, contaminants and other variables.

The durability is also linked to the chemical and physical characteristics of the system, e.g. the type of binder, the dry film thickness. These CUI related performance characteristics can be evaluated by artificial tests. Resistance to water or moisture, boiling water, steam interface, electrolytes in the system, thermal exposure, thermal shock, and thermal cycling are of primary interest.

Artificial tests and durations specified in this document have been selected to help ensure that potential coating systems will have the characteristics needed for the durability required in the intended service. Results from artificial tests should be used with caution, because artificial testing will not necessarily have the same effect as natural exposure. Many factors have an influence on the progress of degradation and, in the laboratory, it is not possible to accelerate all of them using the most effective method. It is therefore difficult to make a reliable ranking of coating systems of very different compositions from artificial tests in the laboratory. This can sometimes lead to efficient protective coating systems being rejected because they cannot pass these tests.

4.2 Laboratory tests

As CUI environments are very specific and have special requirements, several tests are included so that coating products can exhibit performance in harsh environments typical of CUI exposure. These include thermal performance, boiling water, thermal shock, thermal cycling, peak temperature performance, and long term isothermal conditions. In addition, these coating products shall provide corrosion protection for long periods of time at ambient conditions, and in possibly wet conditions related to initial coating application prior to process start up, time associated with process shutdowns, and short term mothballing of the facility.

Standard weathering testing procedures shall be used to establish ambient related corrosion control test procedures and acceptance criteria. Both air dried and conditioned test samples and heat conditions test samples shall be evaluated.

Additional CUI and high temperature related tests shall also be used in order to verify the ability of a coating to work under insulation at the prescribed conditions.

Inorganic zinc primers or other sacrificial coatings are no longer recommended in CUI environments due to the accelerated corrosion related to wet environments. If testing and acceptance is required, additional testing, as agreed between the interested parties, can be performed. However, long term wet environments are difficult to accelerate and as such the specifier/owner should be careful with any acceptance criteria for sacrificial coatings.

4.3 Additional laboratory tests

Other test methods may also be used by agreement between interested parties.

5 CUI classification environments

<u>Table 1</u> provides a list of CUI classification environments including the minimum and maximum temperatures for all environments. These descriptions include both isothermal and cyclic conditions.

 Classification
 Minimum temperature
 Peak temperature

 CUI-1
 -45 °C
 to 60 °C

 CUI-2
 -45 °C
 60 °C to 150 °C

 CUI-3
 -45 °C
 150 °C to 204 °C

Table 1 — CUI classification environments

Further, each coating should be qualified for each specific CUI classification where it is intended to be used. A coating that meets the requirements of a CUI-1 classification does not necessarily meet the requirements of a CUI-3 classification, and a coating that meets the requirements of a CUI-3 classification does not necessarily meet the requirements of a CUI-1 classification. By consolidating testing some tests can be used for more than one classification.

For insulated service for temperatures above 204 °C, additional testing may be performed as agreed by interested parties.

An optional classification for cryogenic cycling exposure ("-Cryo") may be added to each of the classifications in <u>Table 1</u>, when cryogenic testing and acceptance are included. In such cases the classifications as presented in <u>Table 2</u> are appropriate.

ClassificationMinimum temperaturePeak temperatureCUI-1-Cryo-196 °Cto 60 °CCUI-2-Cryo-196 °C60 °C to 150 °CCUI-3-Cryo-196 °C150 °C to 204 °C

Table 2 — CUI classification cryogenic environments

6 Test samples

6.1 Test panels <

This document requires the use of test panels and other testing surfaces that are available as standard shapes typically available on the market place. Both A-36 or S275 carbon steel and 316 (316L) austenitic stainless steel test panels shall be incorporated in testing and also shapes as described in 6.2 to 6.7.

6.2 Steel substrates

Test panels will be as follows unless otherwise agreed to and documented.

6.2.1 Carbon steel test panels shall be made of A-36 or S275 carbon steel. The minimum panel size shall be 3 mm \times 150 mm \times 70 mm or as agreed and documented by the parties. The thickness of the test panels shall not allow for bending as a result of heating and quenching. Unless otherwise agreed, the panel surface shall be prepared by abrasive blast-cleaning to meet the requirements of the corresponding technical product data sheet as per the coating manufacturer's instructions. In all other respects, test panels shall comply with ISO 7384.

- **6.2.2** Austenitic stainless steel test panels shall be used when stainless steel panels are specified. The grade shall be 316 or 316L stainless steel or as agreed and documented by the parties. The minimum panel size shall be 3 mm × 150 mm × 75 mm. Unless otherwise agreed, the panel surface shall be prepared by abrasive blast-cleaning to meet the requirements of the corresponding technical product data sheet as per the coating manufacturer's instructions. In all other respects, test panels shall comply with ISO 7384.
- **6.2.3** Square carbon steel tubing (A-36, ASTM A-500, or S275) ASIC standard shape HSS4X4X1/4 measuring approximately 101,4 mm by 101,4 mm, 406,3 mm long with a wall thickness of 6,35 mm shall be used for the multi-phase CUI cyclic test. Each coating sample area shall be a minimum of 101,4 mm long, on all four sides of the square tube with just the front face scribed with an X-cut. Each tube shall have an endcap welded on each end with a 25,4 mm pipe 101,5 mm long with a threaded connector. The two pipes shall be centred horizontally and shall be vertically attached with a centre 25,4 mm down from the top of the square tube. The completed tube should be tested to ensure that the welds do not leak. Figure 2 provides a general layout (see 7.4.2). Unless otherwise agreed, the tube's exterior surfaces shall be prepared by abrasive blast-cleaning to meet the requirements of the corresponding technical product data sheet as per the coating manufacturer's instructions.
- **6.2.4** A standard black carbon steel pipe (A-36 or S275), approximately 600 mm long, with 50 mm outside diameter with typical wall thickness of 5 mm shall be used for the optional vertical insulation pipe test. Unless otherwise agreed, the test surface shall be prepared by abrasive blast-cleaning to surface preparation to meet the requirements of the corresponding technical product data sheet as per the coating manufacturer's instructions.

6.3 Sampling of coatings

A representative sample of the product to be tested (or of each product in the case of a multi-coat system) shall be taken in accordance with ISO 15528. Each sample for testing shall be examined and prepared in accordance with ISO 1513.

6.4 Number of test panels

At least three panels shall be prepared for each test, unless otherwise specified. Each tube shall have the coating applied three times with the fourth space for a control coating.

6.5 Coating systems

6.5.1 Coating application

The test panels/surfaces shall be dry and free of dust, grease and any other foreign matter, immediately prior to coating application and in keeping with the coating manufacturer's recommendations. The test panels/surfaces shall be coated (preferably by spraying), air dried and cured in strict accordance with the coating manufacturer's recommendations. Each coat shall be homogenous in thickness and appearance and free from runs, sags, misses, pinholes, wrinkling, gloss variation, cissing, particle inclusions, dry overspray and blistering. If the manufacturer's drying requirements are in conflict with <u>6.5.4</u>, the requirements of <u>6.5.4</u> shall take precedence, unless agreed to by all parties. Appropriate protection shall be applied to the edges and back side of test panels.

6.5.2 Dry film thickness

The method and procedure for checking dry film thickness shall be in accordance with ISO 19840 for rough surfaces. After each coat is sufficiently hardened, the dry film thickness of the applied coating shall be measured on the test surface of the test panel/surface at five locations (in the centre and each corner, at least 15 mm to 20 mm from the panel edge or rounded surface of the square tube) and these measurements shall be recorded as the minimum, arithmetic mean and maximum. The dry film thickness shall be within the range of the manufacturer's specifications and shall not exceed 20 % of the specified value.

6.5.3 Overcoating interval

For each layer of coating application, the overcoating interval shall comply with the coating manufacturer's recommendations.

6.5.4 Conditioning

Unless otherwise agreed, the coated test samples shall be conditioned for three weeks in a standard atmosphere of 23 °C \pm 2 °C with 50 % \pm 5 % relative humidity or 20 °C \pm 2 °C with 65 % \pm 5 % relative humidity, as defined in ISO 554, before testing, and in accordance with the requirements of the corresponding technical product data sheet as per the coating manufacturer's recommendations.

6.5.5 Heat conditioning

Heat conditioning of the test samples shall be performed on applied and conditioned test samples. Heat conditioning shall consist of heating the conditioned test panels to the maximum temperature of the classification for 20 h in a muffler oven or other similar device. The test panels shall then be removed and air cooled for 4 h. This process shall be repeated a total of 5 times, providing for a total of 100 h exposure at maximum temperature of the classification and 20 h of air cooling time.

6.6 Scribe

If a scribe is required for testing of coatings on steel substrates, the scribe shall be in accordance with ISO 12944-6. Special care should be taken to ensure that potentially hot and small metal fragments do not affect the sample. Individual test procedures will indicate the need of a scribe for testing.

6.7 Reference system

It is recommended to use a coating system that has been in successful use for years on site and whose performance as indicated by laboratory testing is well known, as a reference system. This system shall be as similar as possible in composition and/or generic type and dry film thickness to the coating system being tested.

7 Test procedures and assessment

7.1 Assessment and acceptance

All tests shall be conducted in triplicate. At least two of the three tests shall comply with the requirements specified in this document. Triplicate for the multiphase CUI cycle test shall be three separate tubes run in three separate test cycles. Triplicate for the optional vertical pipe test shall be three separate tubes.

7.2 Assessment of adhesion and artificial ageing

7.2.1 Adhesion testing before artificial ageing

The applied and conditioned coating sample panels and the heat conditioned coating sample panels shall be tested for adhesion. This includes both the carbon steel test panels and the stainless steel test panels. Table 3 provides list of testing and acceptance criteria.

Table 3 — Adhesion assessment before artificial ageing for carbon steel and stainless steel test panels

Assessment method	Requirement	Conditions
ISO 2409	Classification 0 to 2	If the dry film thickness of the coating system is less than or equal to 250 $\mu \text{m}.$
		In case of cross cut value of 2, a pull-off test in accordance with ISO 4624 shall be conducted
ISO 4624	No adhesive break between steel and the primer (first coat) (unless pull-off values are 5 MPa or more)	ატ

7.2.2 Artificial ageing

<u>Table 4</u> provides test methods and durations as required for each classification. <u>Table 5</u> provides acceptance criteria for these tests. <u>Annex A</u> provides a rationale for the testing of ambient and heat conditioned test panels.

Table 4 — Ambient test methods and duration for carbon steel test panels

	ISO	9227	ISO 2	812-2
	(neutral s	salt spray)	(water in	nmersion)
Classification	Duration		Duration	
	Scr	ribed	Scribed	
	Applied and conditioned	Heat conditioned	Applied and conditioned	Heat conditioned
CUI-1	720 hours	480 hours	3 000 hours	2 000 hours
CUI-2	720 hours	480 hours	3 000 hours	2 000 hours
CUI-3	720 hours	480 hours	3 000 hours	2 000 hours

Table 5 Ambient corrosion test acceptance criteria

Test method	Classificati	ns	ISO 4628-2	ISO 4628-3	ISO 4628-4	ISO 4628-5	ISO 4628-8
ISO 9227	CUI-1 €UI-2	CUI-3	0 (S0)	Ri 0	0 (S0)	0 (S0)	2/3
ISO 2812-2	CUI-1 CUI-2	CUI-3	0 (S0)	Ri 0	0 (S0)	0 (S0)	2/3
Time of assessment (after end of test)				Immed	liately		<8 h

7.2.3 Adhesion testing after artificial ageing

The artificially aged test panels shall be tested for adhesion after the prescribed duration (time of assessment) in the standard atmosphere in accordance with ISO 554. This includes only the carbon steel test panels. Table 6 provides a list of testing and acceptance criteria.

Table 6 — Adhesion assessment after ambient corrosion testing for carbon steel test panels

Assessment method	Requirements	Time of assessment	Conditions/Remarks
ISO 2409	Classification 0 to 2	Assessment after 12 d in standard atmosphere as defined in ISO 554	If the dry film thickness of the coating system is less than or equal to 250 μ m.
			In case of cross cut value of 2, a pull-off test in accordance with ISO 4624 shall be conducted
ISO 4624	No adhesive break between steel and the primer (first coat) (unless pull-off values are 5 MPa or more)	Assessment after 12 d in standard atmosphere as defined in ISO 554	2018

7.3 Thermal cycling test

The test panel is then placed in the oven and heated. When the test panel reaches the maximum temperature of the classification, it shall immediately be dipped/quenched into ice water covering at least 3/4 of the test panel and left immersed until the temperature of the test sample metal temperature is reduced to <15 °C. This shall be repeated for the number of cycles required. The coated surface shall then be evaluated. Minimum and maximum thermal cycling temperatures and the number of cycles are provided for each CUI classification in Table 7. Acceptance criteria are provided in Table 8.

Table 7 — Thermal cycling test for carbons steel test panels

Classification	Minimum temperature	Maximum temperature	Cycles
CUI-1	5 °C	60°C	20
CUI-2	5 °C ⋅	150 °C	20
CUI-3	5 °C	204 °C	20

Table 8 — Thermal cycling acceptance criteria

Classifications	ISO 4628-2	ISO 4628-3	ISO 4628-4	ISO 4628-5
CUI-1	0 (50)	Ri 0	0 (S0)	0 (S0)
CUI-2	C0 (S0)	Ri 0	0 (S0)	0 (S0)
CUI-3	0 (S0)	Ri 0	0 (S0)	0 (S0)

7.4 Multi-phase CUI cyclic corrosion test

7.4.1 Description of test

The multi-phase CUI cyclic corrosion test process is used to test the ability of the coating to work in typical environments where CUI occurs. This test process includes dry heat, thermal shock, immersion, boiling water, steam interface, and shut down time. This test takes six weeks and does not consider the effect related to individual insulation materials. This test process is used for CUI-2 and CUI 3, as these CUI coatings extend into the boiling water range.

7.4.2 Test equipment-apparatus

The multi-phase cyclic CUI test method is designed to provide an overall test procedure for coatings for elevated temperature insulated service. It includes cyclic testing methods providing dry heat, intermittent boiling water for an extended period of time, a steam interface, and an ambient state where there is no heating.

This test process does not take into account the effect of any possible detrimental chemicals given off by specific insulation materials. The immersion and boiling water portion of this test utilizes water with 5 % NaCl (m/v).

To understand the effect of any chemicals associated with the use of any particular insulation, a specific solution can be agreed between the interested parties. Any change of solution shall be documented in the final report.

Figure 1 provides an overall schematic diagram of the equipment and guidance as to the test set up.

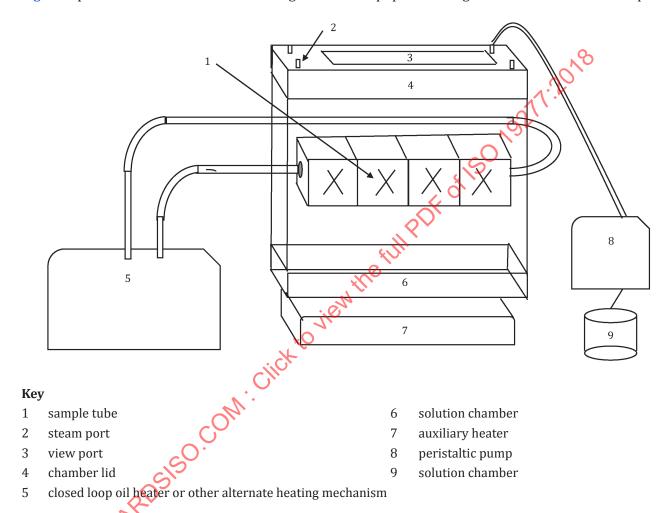


Figure 1 — Schematic presentation of multi-phase CUI cyclic corrosion test

The apparatus includes the following items.

- a) Closed-loop oil heater with piping of approximately 2,54 mm internal diameter connecting to the CUI sample tube. The heating unit should be capable of keeping the circulating heating oil at the specified testing temperature. The heater is typically three phase with 6 kW heating capacity. Alternate heating systems are acceptable, if the temperature of the surface of the sample tubes is maintained with ±5 °C.
- b) Closed-loop heat transfer fluid/oil for operating to the maximum temperature of the test in the range of 150 $^{\circ}$ C to 175 $^{\circ}$ C can be used.
- c) CUI sample tube, coatings applied and conditioned in accordance with <u>6.5.4</u>. Figure 2 provides a schematic presentation of the CUI sample tube. Each coating tested shall have a least a 100 mm wide continuous application on all four sides of the tube.

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- d) Solution chamber and lid with view port and with four 6,35 mm ports: three for steam release and one for tubing connection from the peristaltic pump. Figure 3 provides a schematic presentation of the solution chamber.
- e) Peristaltic pump for pumping the solution to the solution chamber with tubing for transfer of the solution.
- f) Auxiliary hot plate heater typically two heater 220 V 240 V, 5 kW, with approximate dimension 375 mm × 625 mm.

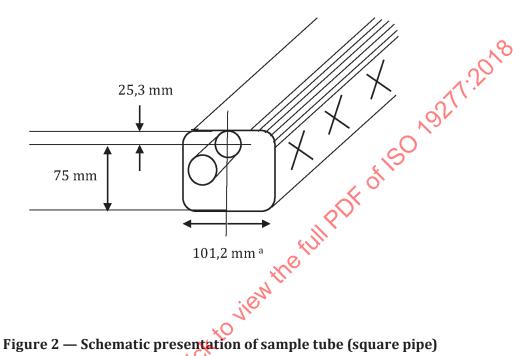
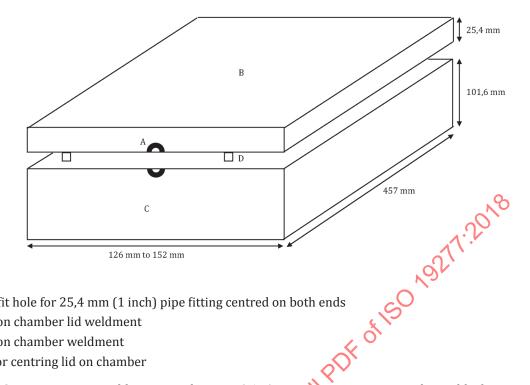


Figure 2 — Schematic presentation of sample tube (square pipe)

Key

Centred.



Kev

- Α loose fit hole for 25,4 mm (1 inch) pipe fitting centred on both ends
- В solution chamber lid weldment
- C solution chamber weldment
- tabs for centring lid on chamber

Components are weldments made using 3,175 mm non-rusting material suitable for temperature of NOTE test protocol. Water tight welds with loose fit between solution chamber and lid.

Figure 3 — Schematic presentation of solution chamber

Test set-up and preparation 7.4.3

The applied and conditioned CUI sample coated tube shall be scribed in accordance with <u>6.6</u> on the face of the square tube as depicted in Figure 2. Note that the pipe fittings are welded offset in one direction. The CUI sample tube should be piped to the closed-loop oil heater using a section of flexible (typically flexible for ease of assembly) stainless tubing to each side of the CUI sample tube.

The solution chamber shall be placed on the auxiliary heater. The CUI sample tube should be placed in the solution chamber with the offset hanging down, so that during the immersion phase the solution will reach the midpoint of the scribe face/side. The CUI sample tube should not be in contact with the bottom or sides of top of the solution chamber. The solution chamber lid shall be placed within the clips of the solution chamber.

The peristaltic pump shall be connected with one of the four steam vents to allow a predetermined amount of solution into the solution chamber.

7.4.4 Test procedure

The test procedure shall be in accordance with the following cycle.

- 1) The tube shall be heated, internally, to the test temperature by heating the oil in the closed-loop oil heater to a temperature that results in the surface of the coated tube reaching 150 °C ± 5 °C or 177 °C ± 5 °C, depending on requirement. The surface temperature of the coatings samples on the square tube will reach test temperature within 30 min to 60 min.
- 2) The oil temperature shall remain at set temperature throughout the heating process, which shall last for 120 h each week. The initial heating process each week will take 30 min to 40 min depending on test temperature requirements. The oil heater will be initially set to 93 °C and once it comes to temperature, left to stabilize for 10 min. The temperature setting is then increased to 150 °C and

left to develop steady state for 10 min. The heater will then be adjusted to 175 °C as can be required for CUI-3.

- 3) After 4 h of the initial dry cycle, the predetermined amount of liquid test media [distilled or deionized water with 5 % NaCl (m/v)] shall be pumped into the enclosure to height where the bottom of the tube is covered on the bottom and up to a height of 50 mm. The peristaltic pump setting and tube selection shall be selected to deliver the solution within 3 min to 4 min. Actual fluid pump rate and volume should be established for each solution chamber size and assemble before the first actual test cycle. This is the initial immersion boiling water cycle.
- 4) With the heat supplied by the heated tube sample as well as the auxiliary heater below the enclosure, the water should boil and evaporate and leave the chamber dry between 3 h and 15 min and 3 h and 45 min. The water will boil vigorously and the water level will evaporate quickly so that typically the tube is no longer immersed after one hour. Adjustment of auxiliary heater set point temperature can be required to adjust the evaporation time frame.
- 5) The chamber shall then be left to run dry and hot for 4 h.
- 6) This shall be followed by a wet cycle using distilled water only for 4 h. The remaining immersion boiling water cycles within the 120 h period shall be with distilled water only, so as not to build up the concentration over 5 % NaCl (m/v).
- 7) These 8 h cycles shall be repeated for the duration of 120 h or 5 d. The last of these cycles will be a wet cycle that shall finish dry.
- 8) The oil heater and the auxiliary heater are shut off for 48 h, representing a standard process shutdown. The tube should be removed and the interior of the chamber and the exterior of the tubes should be rinsed with tap water (squirt bottle, faucet, and/or garden hose) to remove any visible salts, rust or other materials from the chamber. The tube and chamber are left unheated for 48 h. The processes in steps 1) to 8) run again for the next 7 d, and then repeated for a total of 6 times.
- 9) After 6 weeks of testing is completed, the tube shall be thoroughly cleaned and prepared for immediate evaluation within 24 h of completion of the test.

<u>Table 9</u> provides the criteria for the multi-phase cyclic CUI test setup.

Table 9 — Multi-phase cyclic CUI test criteria for carbon steel tube test samples

	Multi-phase cyclic CUI				
Classification	Duration Temperature		Cycles		
	Carbon steel with scribe on vertical side				
	70,		As applied and conditioned		
CUI-2	1 008 h	150 °C	15 cycles consisting of 4 h hot dry environment, 4 h hot and wet environment, followed by 48 h ambient environment, with entire cycle repeated a total of 6 times		
CUI-3	1 008 h	175°C	15 cycles consisting of 4 h hot dry environment, 4 h hot and wet environment, followed by 48 h ambient environment, with entire cycle repeated a total of 6 times		

7.4.5 Acceptance

The sample surface shall be evaluated as soon as possible on the top face (condensation face), scribe face (immersion and steam interface) and the bottom, including the curved corners (intermittent immersion surface). Edges that are 15 mm from the end of the tube, or adjacent to other coatings applications, shall be disregarded. Table 10 provides the acceptance criteria for the multi-phase cyclic test.

Table 10 — Acceptance criteria for multi-ph

Classification	Multi-phase cyclic CUI test temperature	ISO 4628-2	ISO 4628-3	ISO 4628-4	ISO 4628-5	ISO 4628-8
CUI-2	150 °C	0 (S0)	Ri 0	0 (S0)	0 (S0)	2/2
CUI-3	175 °C	0 (S0)	Ri 0	0 (S0)	0 (S0)	2/2

8 Optional tests

8.1 Optional cryogenic cycling testing

8.1.1 General

Coatings meeting the requirements of the classification CUI-1 and CUI-2 may be tested additionally for acceptance for use in cryogenic cyclic service. Maximum warm temperature shall be as a result of the CUI classification and the "-Cryo" suffix can be added to the original CUI classification to indicate that the coating not only passed the requirements of the CUI classification, but also those of cyclic cryogenic service.

8.1.2 Cryogenic test

Prepared 316 or 316L stainless steel test panels or as agreed by the parties shall have coating applied, conditioned and heat conditioned in accordance with 6.5.4. Test panels shall be placed in de-ionized water immersion service for 24 hours, then into the cryogenic freezer or bath at $-190\,^{\circ}\text{C} \pm 20\,^{\circ}\text{C}$ for 1 h, and then placed immediately into to water at the maximum of the CUI classification with the hottest water being boiling water at 100 °C for 1 min. This cycle should be repeated 5 times. Table 11 shows the temperatures and cycles and Table 12 provides the acceptance criteria. This test may be performed for other maximum temperature ranges as agreed between the parties.

Table 11 — Cryogenic cycling test of stainless steel test panels

	Minimum temperature	Water temperature	Cycles		
Classification	316 or 316L stainless steel or as agreed on by the parties				
	Conditioned and heat conditioned				
CUI-1-Cryo	−196 °C	60 °C	5		
CUI-2-Cryo	−196 °C	100 °C (boiling water)	5		
CUI-3-Cryo	−196 °C	100 °C (boiling water)	5		

Table 12 — Cryogenic test acceptance criteria

Classification	Cryogenic test	ISO 4628-2	ISO 4628-3	ISO 4628-4	ISO 4628-5
CUI-1-Cryo	–196 °C to 60 °C	0 (S0)	Ri 0	0 (S0)	0 (S0)
CUI-2-Cryo	–196 °C to 150 °C	0 (S0)	Ri 0	0 (S0)	0 (S0)
CUI-3-Cryo	–196 °C to 204 °C	0 (S0)	Ri 0	0 (S0)	0 (S0)

8.1.3 Adhesion testing after cryogenic test aging

The artificially aged test panels shall be tested for adhesion after the prescribed duration (time of assessment) in the standard atmosphere in accordance with ISO 554. <u>Table 13</u> provides list of testing and acceptance criteria.

Table 13 — Adhesion assessment after cryogenic testing of carbon steel test panels

Assessment method	Requirements	Time of assessment	Conditions/remarks
ISO 2409	Classification 0 to 2	Assessment after 12 d in standard atmosphere as defined in ISO 554	If the dry film thickness of the coating system is less than or equal to 250 μ m.
			In case of cross cut value of 2, a pull-off test in accordance with ISO 4624 shall be conducted
ISO 4624	No adhesive break between steel and the primer (first coat) (unless pull-off values are 5 MPa or more)	Assessment after 12 d in standard atmosphere as defined in ISO 554	2018

8.2 Optional vertical pipe test

8.2.1 Application

The optional vertical pipe test can be performed in addition to the other tests required by this document.

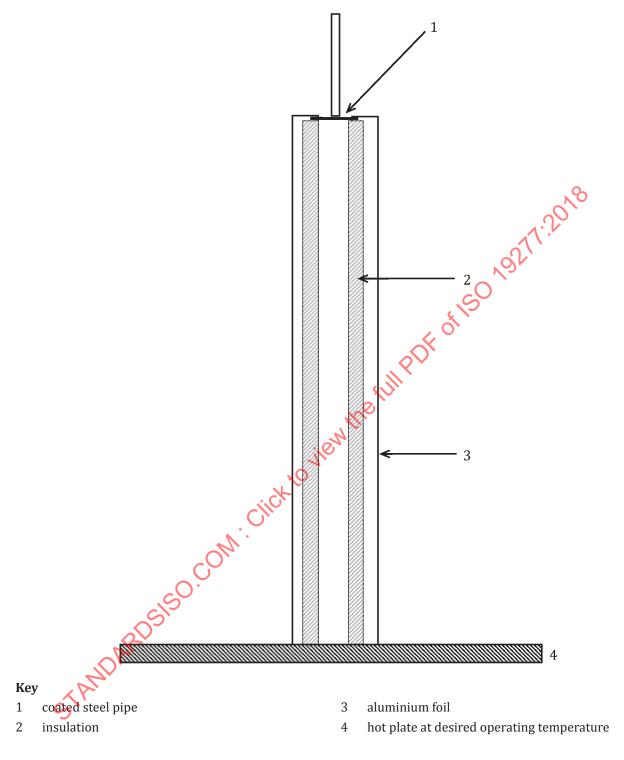
8.2.2 Description of test

The optional vertical pipe test has been designed to test the resistance of coatings to accelerated CUI conditions including ingress of salt solution into the insulation, temperature cycling to high operating temperatures as well as periods of "shutdown" where the pipe cools to ambient temperature.

8.2.3 Test equipment — Apparatus

Figure 4 provides an overall schematic diagram of the equipment and guidance as to the test setup. A general layout of the equipment needed for setting up the vertical pipe/insulation test is as follows:

- a) coated carbon steel pipe: length 600 mm, diameter 60 mm and wall thickness 5 mm;
- b) hotplate for temperatures up to 500 °C;
- c) insulation material (600 mm×50 mm inside diameter × 50 mm deep) with aluminium foil covering to prevent drying out during test;
- d) thermometer compatible with thermocouples to measure the surface temperature profile of the pipe during testing.



 $Figure \ 4-Schematic\ presentation\ of\ vertical\ pipe\ test$

<u>Table 14</u> provides cycles and hot plate set temperature for the optional vertical pipe test.

Table 14 — Optional vertical pipe insulation test for carbon steel pipe or tube samples

	Optional vertical pipe insulation test cycles and settings				
Classification	As applied an	d conditioned			
	Cycles	Hot plate setting			
CUI-2	30	500 °C			
CUI-3	30	500 °C			

8.2.4 Test process scope

Testing shall be carried out on a coated carbon steel pipe of length 600 mm, diameter 60 mm and wall thickness 5 mm. The applied dry film thickness shall be recorded using the sheets in Annex D. The pipe shall be insulated with a specific insulation material and then covered with a waterproof outer cover to prevent the insulation from drying out during the test. The outer cover should be held in place by an appropriate method to keep the insulation in position. Suppliers will often supply insulation with a layer of waterproof outer covering already in place.

8.2.5 Temperature profile calibration

The profile is determined by measuring the temperature using thermocouples attached to the pipe at regular intervals. Temperature measurements shall be done at distances of 50 mm, 100 mm, 150 mm, 200 mm, 250 mm, 300 mm, 350 mm, 400 mm, 450 mm, 500 mm and 550 mm from the hotplate.

8.2.6 Test equipment — Procedure

The pipe should be evaluated as follows.

- 1) Place insulated pipe in a vessel (typically a 10 l tin) and pour 1 l of 1 % NaCl (m/v) solution into the insulation. Allow it to stand for 10 min.
- 2) Place the pipe onto a hotplate (using fume cupboard when necessary), set it to the desired temperature and switch on to the desired temperature. The desired temperature would ensure that the pipe with insulation installed will reach the maximum temperature of the category at least 100 mm above the hotplate.
- 3) Heat the pipe for 8 h. Remove the pipe from the hotplate and place in an appropriate vessel, pour 1 l of 1 % NaCl (m/v) and distilled water solution into the insulation then leave overnight to cool.
- 4) Repeat the cycle above for 5 d per week for 6 weeks (total 30 cycles).
- 5) Evaluate coating degradation at 11 regular intervals around the pipe/tube ignoring the bottom 50 mm (due to damage resulting from the method of pipe termination).

<u>Table 15</u> provides the acceptance criteria.

Table 15 — Optional vertical pipe insulation test acceptance criteria

Classification	ISO 4628-2	ISO 4628-3	ISO 4628-4	ISO 4628-5
CUI-2	0 (S0)	Ri 0	0 (S0)	0 (S0)
CUI-3	0 (S0)	Ri 0	0 (S0)	0 (S0)

8.3 Additional performance tests

Additional tests are recommended, if it is considered necessary to assess the ability of a coating system to provide corrosion protection, and as agreed between the parties. If more information is needed on corrosion protection behaviour, and/or the service conditions are outside of those established by the category or classification, additional test methods may also be used by agreement between the interested parties.

9 Test report

The test report shall contain at least the following information:

- a) the test laboratory (name and address);
- b) the date of each test;
- c) a description of the substrate and substrate surface preparation;
- d) all details necessary to identify the protective coating system (manufacturer, names or reference numbers of the products, batch numbers, numbers of coats, dry-film thickness for each coat);
- e) all details necessary to identify the reference system;
- f) the duration and conditions of drying/curing and conditioning;
- g) the classification of the coating system according to the test results obtained;
- h) the tests carried out and the duration of each test;
- i) the results for each test panel;
- j) any deviation from the test methods specified.

The test report shall explicitly state that the test equipment and procedure were in accordance with the relevant ISO standard.

The test report shall be signed by the person performing the tests and by the laboratory manager or by another authorized representative of the laboratory. Examples of test report forms are given in Annexes B to D.

Annex A

(informative)

Corrosion testing of conditioned and heat conditioned test samples

The rationale behind selection of corrosion testing methods, duration and acceptance criteria is taken in part from ISO 12944-1, ISO 12944-2 and ISO 12944-6. The ISO 12944 series describes test methods, durations and acceptance criteria for coatings for typical atmospheric service. ISO 12944-1 describes three durability ranges based in years until first maintenance. ISO 12944-2 describes classifications of environments based on various ambient environments based on typical corrosion rates. ISO 12944-6 describes testing and acceptance criteria.

For corrosion testing within this document, two conditions for the coating exist as a result of the heated and insulated environment.

Condition 1

This condition would be that the coatings are as applied and have not gone through a heating cycle. This condition would be found as part of a new construction project, where the coatings are applied at some stage of fabrication and construction of the facility and then may not be put into service for a short to extended period of time, depending on the construction schedule. With the complexity and size of modern construction this could extend to multiple years.

A C5-M environment and IM-3 (for immersion risk) with high durability life were selected for evaluating coating performance for this condition.

Condition 2

This condition would be that the coatings have been applied and put into service\heated and then the thermal conditions are changed or reduced to where a corrosion cell can develop. This would be typical of a temporary shutdown, outage, and even a short term mothballing of the facility. In essence, this is a much shorter time requirement typically represented by a one month to two month shutdown and possibly a short term mothballing of up to several months.

A C5-M environment and [M-3] (for immersion risk) with medium durability life was selected for evaluating coating performance for this condition.

NOTE For both conditions condensation testing was omitted as immersion service was being used.

Annex B

(informative)

Example of test report for CUI coating acceptance

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Assessment before corrosion testing — Adhesion testing								
ISO 2409 or ISO 4624	≤2 or 5 MPa with cohesive break,	Panel 1	Panel 2	Panel 3	Pass/Fail			
	If ISO 2409 test result in a 2 then also record results of ISO 4624							
	Panel number							
Carbon steel or Stain-	Conditioned							
less steel	Heat conditioned							

For CUI-1, CUI-2, C	UI-3, Neutral salt spray				
Corrosion testing	Requirement	Panel 1	Panel 2	Panel 3	Pass/Fail
	Panel number				
ISO 9227	ISO 4628-2 Blistering – 0(S0)				
Neutral salt spray	ISO 4628-3 Rusting – Ri 0				
	ISO 4628-4 Cracking - 0 (S0)				
	ISO 4628-5 Flaking - 0 (S0)				
	ISO 4628-8 Delamination and corrosion around a scribe – 2/2			-018	
	ISO 2409 after ISO 9227 – ≤2			1.7.	
For CUI-1, CUI-2, C	UI -3, –Immersion testing			J. '	
Corrosion testing	Requirement 3 000 or 500 hours	Panel 1	Panel 2	Panel 3	Pass/Fail
	Panel number		CO		
ISO 2812-2	ISO 4628-2 Blistering – 0(S0)		1/2		
	ISO 4628-3 Rusting – Ri 1	4	0,		
	ISO 4628-4 Cracking - 0 (S0)	20,			
	ISO 4628-5 Flaking – 0 (S0)				

Assessment after corrosion testing — Adhesion testing — After standard environment								
ISO 2409 or ISO 4624	≤2 or 5 MPa with cohesive break, If ISO 2409 test result in a 2 then also record results of ISO 4624	Panel 1	Panel 2	Panel 3	Pass/Fail			
	Panel number							
Carbon steel or	Conditioned							
Stainless steel	Heat conditioned							

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