
**Determination of the resistance
to cryogenic spillage of insulation
materials —**

**Part 1:
Liquid phase**

*Détermination de la résistance des matériaux d'isolation thermique
suite à un refroidissement cryogénique —*

Partie 1: Phase liquide



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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 on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 67, *Materials, equipment and offshore structure for petroleum, petrochemical and natural gas industries*, Subcommittee SC 9, *Liquefied natural gas installations and equipment*.

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Introduction

The test described in the procedure in this document is one in which some of the properties of cryogenic spillage protection materials can be determined. This test is designed to give an indication of how cryogenic spillage protection materials will perform in a sudden exposure to cryogenic liquid.

The dimensions of the test specimen can be smaller than typical items of structure and plant and the release of liquid can be substantially less than that which might occur in a credible event. However, individual thermal and mechanical loads imparted to the cryogenic spillage protection materials, from the cryogenic spillage defined in the procedure described in this document, have been shown to be similar to those by large-scale cryogenic spillage.

Further parts of ISO 20088 are planned for future publication:

- Part 2 : Vapour phase;
- Part 3: High pressure jet release.

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Determination of the resistance to cryogenic spillage of insulation materials —

Part 1: Liquid phase

CAUTION — The attention of all persons concerned with managing and carrying out cryogenic spillage testing is drawn to the fact that liquid nitrogen testing can be hazardous and that there is a danger of receiving a “cold burn” and/or the possibility that harmful gases (risk of anoxia) can be evolved during the test. Mechanical and operational hazards can also arise during the construction of the test elements or structures, their testing and disposal of test residues. An assessment of all potential hazards and risks to health shall be made and safety precautions shall be identified and provided. Appropriate training and personal protection equipment shall be given to relevant personnel.

1 Scope

This document describes a method for determining the resistance to liquid cryogenic spillage on cryogenic spillage protection (CSP) systems. It is applicable where CSP systems are installed on carbon steel and will be in contact with cryogenic fluids.

Liquid nitrogen is used as the cryogenic medium since it has a lower boiling point than liquid natural gas or liquid oxygen and it is not flammable. Additionally, it can be safely used for experiment.

Future parts of the standard will cover vapour phase and jet exposure conditions.

The test laboratory is responsible to conduct an appropriate risk assessment according to local regulation in order to consider the impact of liquid and gaseous nitrogen exposure to equipment and personnel.

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 630-1, *Structural steels — Part 1: General technical delivery conditions for hot-rolled products*

ISO 845, *Cellular plastics and rubbers — Determination of apparent density*

ISO 8301, *Thermal insulation — Determination of steady-state thermal resistance and related properties — Heat flow meter apparatus*

ISO 16903, *Petroleum and natural gas industries — Characteristics of LNG, influencing the design, and material selection*

ISO 22899-1, *Determination of the resistance to jet fires of passive fire protection materials — Part 1: General requirements*

EN 10029, *Tolerances on dimensions, shape and mass for hot rolled steel plates 3mm thick or above*

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:

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

3.1 floating liquid natural gas FLNG

floating liquid natural gas facilities such as floating production storage and offloading (LNG-FPSO), floating storage and re-gasification unit (LNG-FSRU)

3.2 cryogenic spill

unintended exposure to cryogenic liquid (CL) at -196°C

3.3 cryogenic spill protection CSP

coating or cladding arrangement, or free-standing system which, in the event of a cryogenic spill, will provide thermal protection to restrict the heat transfer rate of the substrate

3.4 limiting temperature

minimum temperature that the equipment, assembly or structure to be protected may be allowed to reach

3.5 release point

assembly from which the cryogenic fluid flows out

3.6 sponsor

person or organization who/which requests a test

3.7 specimen owner

person or company that holds/produces a material to test

4 Test configurations

4.1 General

There is one basic configuration under which the test can be conducted. This is a liquid configuration where the material to be tested is rapidly exposed to liquid nitrogen in a pool at a temperature of -196°C . For reasons of clarity, flexible hoses used for fume extraction are not shown in [Figures 1 to 4](#) below.

4.2 Sample holder

Samples will be tested in a sample holder with exact dimension as specified in ISO 22899-1:2007, Figure 11.

5 Construction of the test items and substrates

5.1 General

The key items required for the test are:

- a liquid nitrogen injection point;
- a sample holder;
- the insulation part.

It is important to reduce vapour generation during the liquid nitrogen dumping. Flexible hoses are to be used without forced ventilation.

5.2 Material

The material normally used is a 10 mm thick steel plate complying with ISO 630-1, Grade Fe 430. An all welded construction shall be used and all welds shall be 5 mm fillet and continuous unless otherwise stated. All dimensions are in millimetres and, unless otherwise stated, the following tolerances shall be used:

- whole number $\pm 1,0$ mm;
- decimal to point, 0 mm $\pm 0,4$ mm;
- decimal to point, 00 mm $\pm 0,2$ mm;
- angles 0' 30";
- radius 0,4 mm.

5.3 Release tank

The liquid nitrogen is contained within a tank of a constant surface area either a square base of 750 mm or circular base of 846 mm diameter. The release orifice shall be 100 mm in diameter.

When there is 250 l of stable non-bubbling liquid nitrogen within the tank, the test is ready to start. The release tank shall be constructed of cryogenic resistant stainless steel and externally insulated for personnel protection.

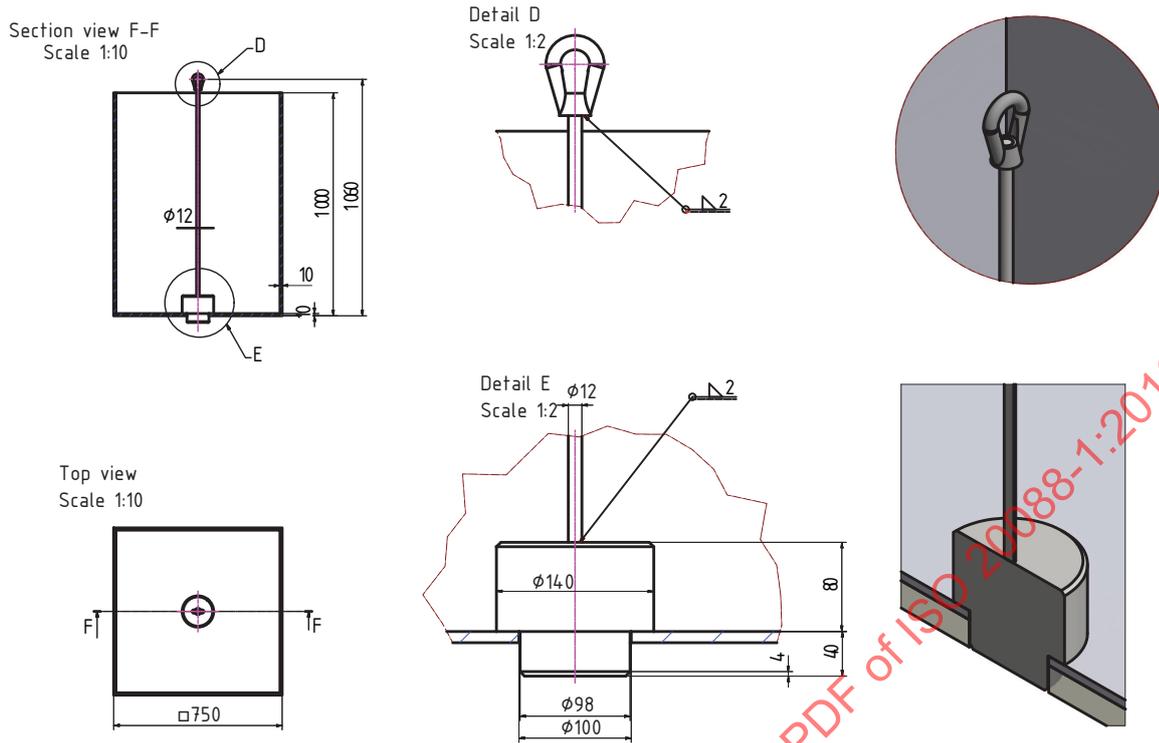


Figure 1 — Layout of a square release tank

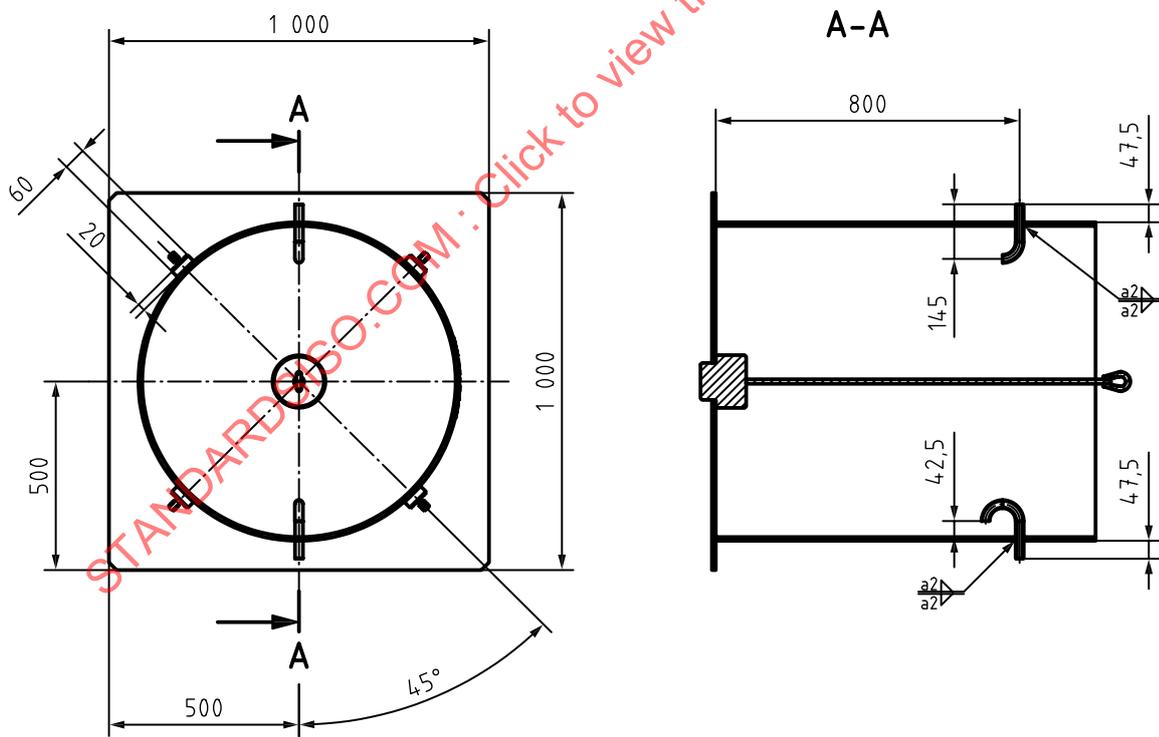


Figure 2 — Layout of a cylindrical release tank

5.4 Specimen support

A square generic support will be used to hold the test sample. It shall be constructed with 10 mm carbon steel. The centre of the table is hollow and will receive the sample holder as shown in [Figure 3](#).

- thickness: 30 mm ± 2 mm;
- thermal conductivity at 20 °C: 40 mW/m·K ± 4 mW/m·K determined as in accordance with ISO 8301.

5.5 Sample holder

All beams should be welded onto the bottom flat panel. Planarity of the bottom panel shall be in accordance with EN 10029 as shown in [Figure 4](#).

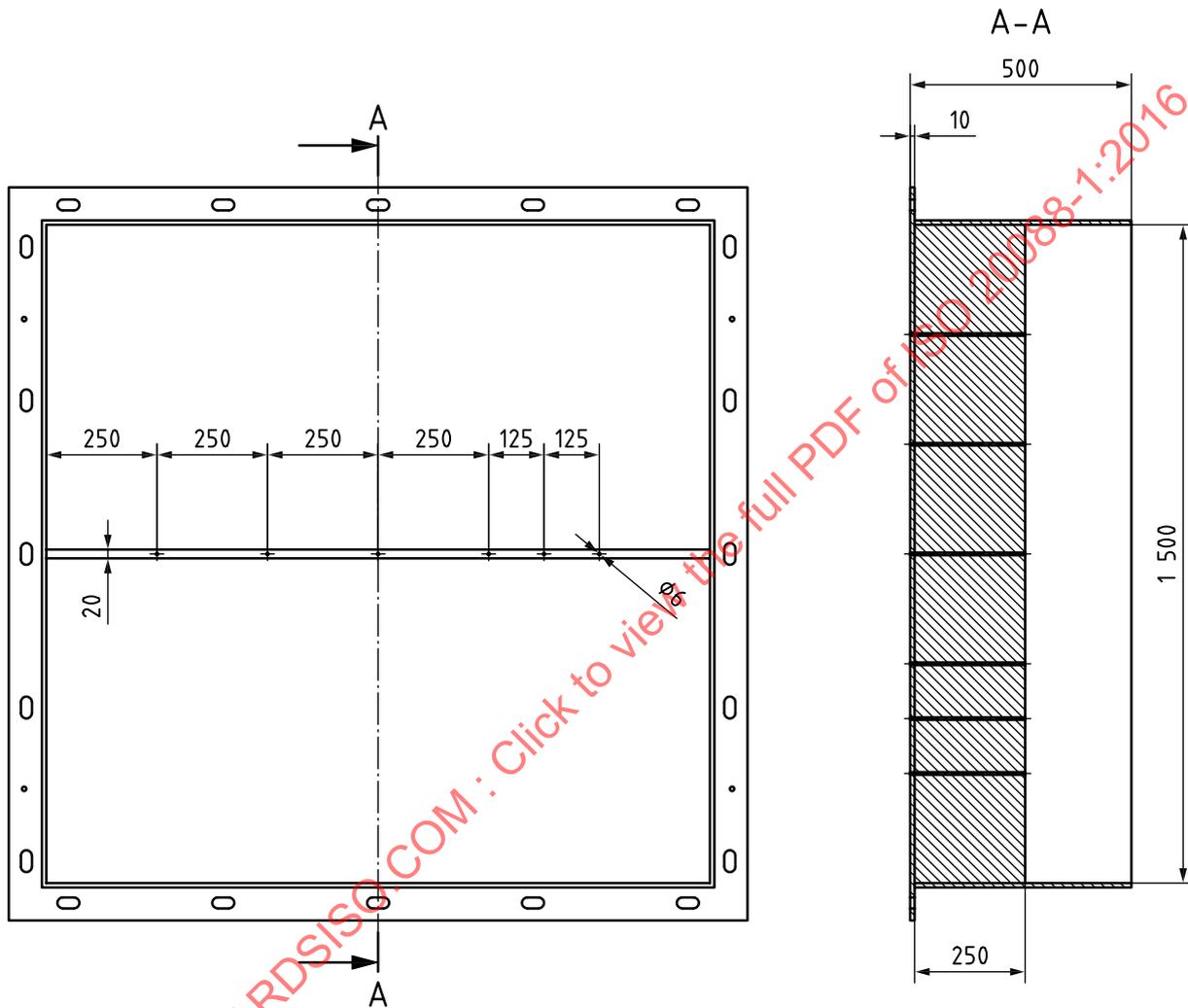


Figure 4 — Sample holder

The sample holder walls and top should be externally insulated with an insulation material as described in [5.4](#). Rigid foam board walls should have a height of 1 m. The box shall be fully vapour tight.

The top cover shall have three holes: one for liquid nitrogen injection in the centre and two for fume extraction (see [Annex B](#)).

5.6 Test method

When there is 250 l of liquid nitrogen within the tank, the test is ready to start. At the beginning of the test the liquid nitrogen is quickly released towards the specimen of material under test from a height of 1 000 mm. It is necessary to ensure a permanent 5 cm level of liquid nitrogen in both parts of the basin in the sample holder for the duration of the test, liquid nitrogen is fed via the tank with suitable cryogenic flexible hose for example with 6,35 mm diameter. The procedure for the test is detailed in [Clause 9](#).

6 Cryogenic spillage protection materials

6.1 General

CSP systems generally come in two forms: wet applied materials/coatings and preformed materials. Preformed systems include boards, tiles, blankets, sandwich panels, etc. and are characterized by systems that include joints and fixings. Pre-formed systems may be used in conjunction with wet applied materials.

The application/installation methodology, including any necessary surface preparation, reinforcement, thickness, top-coats, field joints, etc., is to be determined by the sponsor and/or specimen owner and details provided for inclusion within test report.

6.2 Wet applied coating systems

For testing CSP systems/materials that are wet applied as coatings, the sample holder test specimen, as shown in [Figure 4](#), shall have material applied directly to all internal surfaces. The system thickness should be uniform across the whole specimen.

6.3 Pre-formed system testing

When testing pre-formed systems, the system shall, as a minimum, be installed to cover the bottom of the test assembly on to which the LN₂ pool is formed, as shown in [Figure 4](#).

The method of installing the system shall include representative joints, fixings and wet applied material interface details; a minimum of two joints should be included as follows:

- a) one joint located along line of thermocouples: 4, 7, 10 (or 3, 6, 9) as shown in [Figure 5](#);
- b) one joint located along line of thermocouples: 5, 6, 7, 8 as shown in [Figure 5](#).

Joints should be tested, either in a single test or separate tests as determined by the sponsor and/or specimen owner ensuring the details are representative in accordance with [Clause 10](#).

7 Instrumentation

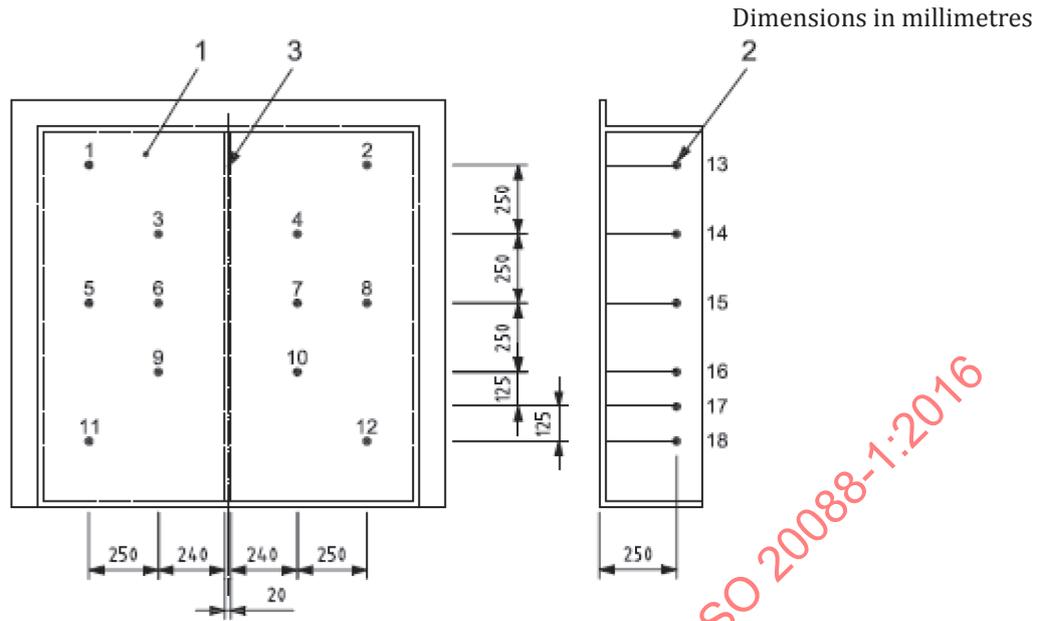
7.1 General

Thermocouples shall be fastened to all test specimens. The type and fixing shall be in accordance with one of the methods described in [Annex A](#).

Readings shall be recorded at intervals of not more than 1 s.

7.2 Thermocouple location

Thermocouples are positioned as shown in [Figure 5](#).



Key

- 1 view from rear of specimen
- 2 thermocouple location
- 3 simulated corner or edge feature of “I” beam

Figure 5 — Thermocouple location at sample holder

8 Test apparatus and conditions

8.1 Injection point and position

8.1.1 General

Details of construction of the injection point, from which the liquid nitrogen is issued, are given in [5.3](#). Liquid nitrogen should be a minimum of 98 % purity.

8.1.2 Injection point position

The injection point shall be positioned vertically above and normal to the bottom panel of the test specimen. The tip of the injection point shall be located $1\ 000\ \text{mm} \pm 5\ \text{mm}$ above the web of the CSP material specimen. The centre of the injection point shall align with the centre of the test specimens.

8.2 Test environment

The test shall be operated either indoor or outdoor. The average sample temperature prior to testing shall be between $23\ \text{°C} \pm 2\ \text{°C}$, if curing and conditioning is conducted under different conditions, it shall be clearly stated in the test report. Ambient temperature shall be within a range from $-10\ \text{°C}$ to $+40\ \text{°C}$.

For outdoor condition, the test shall be carried out in an environment in which the effects of the weather do not significantly affect the test and the following conditions apply:

- sample temperature is $23\ \text{°C} \pm 2\ \text{°C}$ at the beginning of the test;
- outdoor temperature shall be within a range from $-10\ \text{°C}$ to $+40\ \text{°C}$;
- no direct sunlight exposure;

— no rain exposure.

9 Test procedure

The test procedure shall include the following.

- a) The specimen owner shall specify the duration of the test.
- b) 250 l of liquid nitrogen is poured into the release tank.
- c) The test starts when the liquid nitrogen is first released.
- d) Liquid nitrogen shall be fully released within 90 s.
- e) The specimen owner shall provide the specimen for the test in a condition representative of its practical application. Specifically, the test should feature the proposed joint geometry as shown in [6.3](#).
- f) Photographs shall be taken of the test specimen before the test.
- g) If the cryogenic protection is a coating material, the thickness shall be measured at the positions specified in [Figure 6](#). The measurement positions indicated shall be regarded as approximate. If there are clear signs of thinning or thickening at positions away from those indicated for measurement, additional measurements should be taken. When free standing panels are used, calipers shall be used prior to installation.

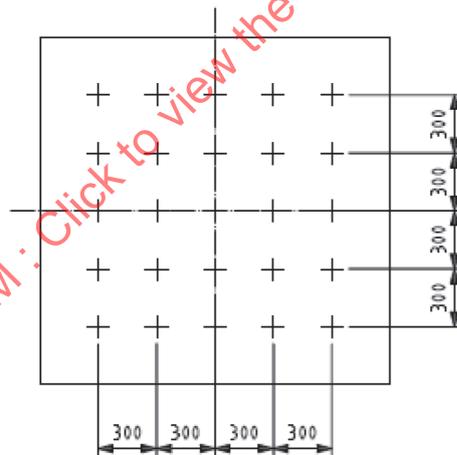


Figure 6 — Thickness measurement points

- h) During the test, a minimum level of 5 cm of liquid nitrogen shall be continuously maintained inside the pool on both sides of the central web. The level shall be monitored by the use of thermocouples in both parts of the sample holder (more detail is provided in [Annex C](#)).
- i) Observations shall be recorded of significant details of the behaviour of the test specimen during the test and after the liquid nitrogen is removed. Information on deformation or partial removal of the surface or cracking shall be noted.
- j) Photographs of the test specimen shall be taken as soon as is practicable after the end of the test. These pictures shall be included in the test report.
- k) Provision should be made that a sample can be inspected within 15 min after termination of the test. Sample access time should be recorded.

10 Repeatability and reproducibility

The test method described in this document has been shown to be repeatable and reproducible between test laboratories. It is the responsibility of the specimen owner to demonstrate that the test results of the sample(s) are representative of their CSP system including any joints and fixing details.

11 Uncertainty of measurement

One of the key factors requiring close control is the constant level of nitrogen in the sample holder. The combined (thermocouples, transmitters, logging system) error of the temperature measurement systems are of the order of $\pm 3,5$ °C. Hence, a tolerance of ± 5 °C is allowed when measuring a temperature drop.

12 Test report

The test report shall include the following information:

- a) the name of the testing laboratory, the date of the test, a unique test reference and report identification;
- b) the names of the sponsor/customer, the manufacturer and the product;
- c) documentation on how and when the test specimen was prepared, details of the application of the CSP material, the name of the applying/installing company;
- d) a complete description of the test specimen, including measurements of the thickness of cryogenic spillage protection material and the hardness, if measured; the mean, standard deviation and range of measurements of thickness should be given as well as details of any joint (if applicable), e.g. position, type, size of overlap and method of fixing;
- e) construction drawings of the CSP system wherever possible;
- f) when appropriate, details of any deviations from the normal test configurations and the reasons for them;
- g) a record of test details and post cryogenic exposure characterization including:
 - 1) ambient conditions;
 - 2) liquid nitrogen level measurement and temperature at least every 1 s throughout the test;
 - 3) for assemblies, a full inspection following the test to validate construction details and assess CSP performance (the assembly should be dismantled so that all components of the system can be checked for cryogenic penetration, integrity and general condition and a visual record made);
 - 4) the test result, shall be given in the following format:
 - the appearance of the test specimen before and after the test and photographs;
 - the behaviour of the test specimen in case of cracking and/or unusual sound;
 - temperature/time graphs and spreadsheets of temperatures at no more than 1 s intervals for each thermocouple;
 - temperature/time and spreadsheet of the average of temperatures of all thermocouples;
 - an optional classification in terms of the type of specimen tested, critical temperature drop and period of resistance in accordance with [Annex D](#).

13 Practical applications of test results

13.1 General

This test is designed to give an indication of how cryogenic spillage protection materials will perform at a sudden exposure to cryogenic liquids. The test specimen was the result of the consideration of the application of insulation to protect steel work against cryogenic exposure of process decks and the main decks of FLNGs. It was realized that decks are not just large flat areas but have considerable structural steel protrusions such as supports for the top-side structure, intersection with top-side structure and combing. As such, the test was designed to ensure liquid coverage on the base of a box with a steel web only partially immersed.

Although the procedure has been designed to simulate some of the conditions that occur in an actual cryogenic spillage as stated above (via the heat transfer determination from the carbon steel through the CSP system by means of a pool of liquid nitrogen) it cannot produce all conditions exactly. The results of this test do not guarantee safety but can be used as element of a cryogenic risk assessment for structures. This should also take into account all the other factors that are pertinent to an assessment of the cryogenic hazard for a particular end use such as chemical resistance towards LNG, weathering, ageing, impact or explosion resistance or smoke production, to name a few.

The relevance of the test set up and results with regards to

- cryogenic spill scenario, and
- location of the insulation system on the structure

shall be the responsibility of the end user. The acceptability of the test, in relation to the end use, can be subject to an independent verification authority [such as a class society (if part of their scope)] or remain at the discretion of the owner (or through delegation to the responsible design engineering company or consortium).

13.2 Performance criteria

13.2.1 General

The purpose of this test method is described in the scope. It is not the purpose of this test method to provide guidance on the acceptability of a particular thickness of CSP for items such as free standing panels, nor the coating or method of assembly to be used.

The criterion of performance, provided by the test, is the minimum time required to reach the limiting temperature. However, the factors in [13.2.2](#) shall also be considered when assessing performance.

13.2.2 Coatings and spray-applied materials

13.2.2.1 Substrate temperature

The temperature time profiles at each measurement position shall be used to determine the minimum temperature at each position reached during the test. The position and time of a sudden decrease in the rate of temperature start, if any, should be recorded as it is indicative of possible failure of the cryogenic spillage protection coating at that point. For the same reasons, the localized minimum temperature shall be reported in conjunction with the nearest cryogenic spillage material thickness measurement.

13.2.3 Systems and assemblies

13.2.3.1 Substrate temperature

The temperature time profiles at each measurement position shall be used to determine the minimum temperature at each position reached during the test. As the liquid nitrogen is applied uniformly over

the specimen, the mean substrate temperature shall be used in the evaluation. The position and time of any sudden decrease in the rate of temperature drop, if any, should be recorded as it is indicative of possible failure of the system/assembly at that point.

13.2.3.2 Loss of containment

The penetration of liquid through any cracks, holes or breaches in joints shall be considered when assessing the integrity of a system. The amount of penetration and condition of the method of fixing can be evaluated in the following terms.

- a) Evidence of passage of liquid through the system with the fixing system ineffective reflected by a localized fast drop in temperature.
- b) Evidence of passage of cold vapour through the system with the fixing system effective.
- c) No passage of liquid/vapour through the system and with the fixing system effective.

If the temperature criterion is met, then a specimen meeting criterion c) clearly provides a wider safety margin than a specimen meeting criterion a). A statement of the criterion that is most appropriate should be included in the report.

13.3 Factors affecting the validity of the test

13.3.1 General

It is inevitable that, during some tests, there is a failure of control, instrumentation or of a seal. These are failures of the test procedure and do not indicate a failure of the test specimen. The most common failures are considered in [13.3.2](#), [13.3.3](#) and [13.3.4](#).

13.3.2 Leakage of the release tank

It can happen that the joint at the cryogenic release point is not perfectly liquid tight. If this is rapidly identified the test should start as soon as possible. If it is the contrary, the sample needs to be discarded and a brand new one should be tested.

13.3.3 Failure of thermocouples

Up to three thermocouples can fail during a test and the test still be considered valid provided that the thermocouple position(s) do not correspond to the area of greatest damage.

Failure of thermocouples directly underneath a joint as shown in [6.3](#) will terminate the test.

13.3.4 Loss of sample integrity/loss of containment

A liquid-tight seal should be maintained between the sample and the sample holder. If the sample loses integrity, liquid nitrogen can break through resulting in external cooling of the steel plate holder as well as the sample itself affecting the heat loss characteristics of the rear of the panel. The severity of the breakthrough and its effect on the measured temperatures should be assessed. The test can still be considered valid but the maximum temperature drop measured on the panel and the time taken to reach it should not be adjusted.

Annex A (normative)

Methods of fixing thermocouples

A.1 General

Chromel/alumel (type K) thermocouples, with conductors of a diameter appropriate to the method of fixing shall be used. Thermocouples shall be attached in accordance with one of the methods given in [A.2](#) to [A.6](#).

A.2 “Quick Tip” attachment

Place the two 0,3 mm diameter thermocouple wires into a metal piece with pre-drilled holes for the wires. Squeeze the metal piece in a special tool into a cylindrical shape, 1,5 mm long and 1,5 mm in diameter, firmly gripping both wires in the process. Drill a 2 mm hole, 2 mm deep, in the position where the thermocouple is to be fixed. Place the joint piece in the hole and use a drift to peen over the metal.

A.3 Capacitive discharge welding

Equipment, suitable for attaching thermocouples to a metal substrate, shall be used. Follow the manufacturer's instructions in preparing the surface and attaching the thermocouple.

NOTE Small pieces of stainless steel shim can be spot welded over the thermocouple to add mechanical strength and prevent stress being placed on the junction.

A.4 Drilling and peening

This method shall be used for attaching sheathed or welded junction thermocouples to a metal substrate. If the thermocouple has separate wires, weld them together. Drill a hole of appropriate diameter in the position in which the thermocouple is to be attached with a maximum depth of 5 mm. Insert the thermocouple and peen over the edges of the hole with a centre punch.

A.5 Adhesive

This method shall be used for fixing thermocouples to non-metal substrates. If the thermocouple has separate wires, weld them together. Fix the thermocouple to the substrate with an epoxy adhesive suitable for cryogenic temperature duty.

A.6 Central web thermocouples (structural steel work test specimen)

This method shall be used for installing 2 mm diameter, stainless steel sheathed thermocouples in the central web. The thermocouple shall be a probe-type thermocouple, and of a suitable length (about 330 mm long is appropriate). The holes in the rear wall of the structural steelwork test specimen leading to the central web thermocouple slots shall have parallel threads, and a thermocouple compression fitting with the same thread, but tapered, shall be fitted to the thermocouple. Install the thermocouple into the slot. Screw the front part of the compression fitting, which has the tapered thread, hand tight into the threaded hole, and then unscrew it one turn. Tighten the rear part of the compression fitting on to the front part with the thermocouple pushed fully into the slot.

Re-screw the front part of the compression fitting into the threaded hole, ensuring that the tip of the thermocouple is in positive contact with the end of the slot.

NOTE Measuring the protruding length provides a check of this.

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