INTERNATIONAL STANDARD

ISO 16111

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Transportable gas storage devices — Hydrogen absorbed in reversible metal hydride

Appareils de stockage de gaz transportables — Hydrogène absorbé dans un hydrure métallique réversible

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Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 16111 was prepared by Technical Committee ISO/TC 197, *Hydrogen technologies*, with participation by Technical Committee ISO/TC 58, *Gas cylinders,* Subcommittee SC 3, *Cylinder design.*

This first edition cancels and replaces ISO/TS 16111:2006, which has been technically revised.

Introduction

As the utilization of gaseous hydrogen evolves from the chemical industry into various emerging applications, such as fuel for fuel cells and internal combustion engines and other specialty hydrogen applications, the importance of new and improved storage techniques has become essential. One of these techniques employs the absorption of hydrogen into specially formulated alloys. The material can be stored and transported in a solid form, and the hydrogen later released and used under specific thermodynamic conditions. This International Standard describes the service conditions, design criteria, type tests, batch tests and routine tests for transportable hydride-based hydrogen storage systems, referred to as “metal hydride assemblies” (MH assemblies). Types of MH assemblies may serve as: fuel cell cartridges; hydrogen fuel storage containers; high-purity hydrogen supplies and others.

Transportable gas storage devices — Hydrogen absorbed in reversible metal hydride

1 Scope

This International Standard defines the requirements applicable to the material, design, construction, and testing of transportable hydrogen gas storage systems, referred to as “metal hydride assemblies” (MH assemblies) which utilize shells not exceeding 150 l internal volume and having a maximum developed pressure (MDP) not exceeding 25 MPa (250 bar). This International Standard only applies to refillable storage MH assemblies where hydrogen is the only transferred media. Storage MH assemblies intended to be used as fixed fuel-storage onboard hydrogen fuelled vehicles are excluded. This International Standard is intended to be used for certification purposes.

2 Normative references

The following referenced documents are indispensable for the application 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 7225, *Gas cylinders — Precautionary labels*

ISO 7866, *Gas cylinders — Refillable seamless aluminium alloy gas cylinders — Design, construction and testing*

ISO 9809-1, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa*

ISO 9809-2, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 2: Quenched and tempered steel cylinders with tensile strength greater than or equal to 1 100 MPa*

ISO 9809-3, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 3: Normalized steel cylinders*

ISO 10297:2006, *Transportable gas cylinders — Cylinder valves — Specification and type testing*

ISO 11114-4, *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents — Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement*

ISO 11119-1, *Gas cylinders of composite construction — Specification and test methods — Part 1: Hoop wrapped composite gas cylinders*

ISO 11119-2:2002, *Gas cylinders of composite construction — Specification and test methods — Part 2: Fully wrapped fibre reinforced composite gas cylinders with load-sharing metal liners*

ISO 14246, *Transportable gas cylinders — Gas cylinder valves — Manufacturing tests and inspections*

ISO 14687, *Hydrogen fuel — Product specifications*

ISO 16528-1, *Boilers and pressure vessels — Part 1: Performance requirements*

UN Recommendations on the Transport of Dangerous Goods. Model Regulations

3 Terms and definitions

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

3.1
absorbed
taken and held through the formation of bonding interactions within the bulk of the material

3.2
burst pressure
highest pressure reached in an MH assembly during a burst test

3.3
design stress limit
total stress loading allowed on the shell wall

NOTE In MH assemblies, the shell design takes into account the gas pressure plus other stresses, such as pressure exerted by expansion of the hydrogen absorbing alloy.

3.4
fuel cell cartridge
article that stores fuel for discharge into the fuel cell through a valve(s) that controls the discharge of fuel into the fuel cell

3.5
fuel cell cartridge
MH assembly, which stores hydrogen for use as a fuel in a fuel cell

3.6
full flow capacity pressure
gas pressure at which the pressure relief device is fully open

3.7
hydrogen absorbing alloy
material capable of combining directly with hydrogen gas to form a reversible metal hydride

3.8
internal component
structure, matrix, material or device contained within the shell (excluding hydrogen gas, hydrogen absorbing alloy and metal hydride)

NOTE Internal components may be used for purposes such as heat transfer, preventing movement of the hydrogen absorbing alloy/metal hydride and/or to prevent excessive stress on the shell walls due to hydride expansion.

3.9
internal volume
water capacity of the shell

3.10
maximum developed pressure
MDP
highest gas gauge pressure for an MH assembly at rated capacity and equilibrated at the maximum service temperature

NOTE The MDP term was specifically selected for MH assemblies to avoid confusion with the MAWP and the service pressure used in other ISO International Standards.

3.11
metal hydride
solid material formed by reaction between hydrogen and hydrogen absorbing alloy

3.12
metal hydride assembly
MH assembly
single complete hydrogen storage system, including shell, metal hydride, pressure relief device (PRD), shut-off valve, other appurtenances and internal components

NOTE 1 The MH assembly extends only to, and includes, the first shut-off valve.

NOTE 2 A fuel cell cartridge is a type of MH assembly.

3.13
normal operating conditions
range of pressures, temperatures, hydrogen flow rates, hydrogen quality, etc., specified for all use and filling operations

3.14
normal service conditions
range of pressures, temperatures, hydrogen flow rates, hydrogen quality, etc., specified for all normal operating, transportation and storage conditions

3.15
pressure relief device
PRD
device intended to prevent the rupture of an MH assembly in the event of overpressure or exposure to fire

NOTE A pressure relief device may be "pressure-activated", set to activate at a certain pressure. Alternatively, a pressure relief device may be "thermally-activated", set to activate at a certain temperature. A pressure relief device may also be both "pressure-activated" and "thermally-activated".

3.16
pressure relief valve
PRV
reseatable PRD

3.17
rated capacity
maximum quantity of hydrogen deliverable under specified conditions

3.18
rated charging pressure
RCP
maximum pressure to be applied to the MH assembly for refilling

NOTE The RCP is not necessarily equal to the equilibrium plateau pressure of the hydrogen absorbing alloy.

3.19
reversible metal hydride
metal hydride for which there exists an equilibrium condition where the hydrogen absorbing alloy, hydrogen gas and the metal hydride co-exist

NOTE Changes in pressure or temperature will shift the equilibrium favouring the formation or decomposition of the metal hydride with respect to the hydrogen absorbing alloy and hydrogen gas.

3.20
rupture
structural failure of a shell resulting in the sudden release of stored energy

3.21
shell
enclosure of any shape (cylindrical, prismatic, cubic, etc.) designed to contain the hydrogen gas, metal hydride and other internal components of the MH assembly

NOTE A shell may be a cylinder, a pressure vessel or other type of container.

3.22
stress level at MDP
sum of all the stresses on the shell wall caused by the metal hydride at rated capacity, hydrogen gas at MDP and any other applicable mechanical loadings

3.23
test pressure
required pressure applied during a pressure test for qualification

4 Service conditions

4.1 Pressures

4.1.1 Maximum developed pressure (MDP)

The MDP shall be determined by the manufacturer from the metal hydride's temperature–pressure characteristics at the maximum service temperature. In no case shall the MDP exceed 0,8 times the test pressure of the shell. The MDP shall not exceed 25 MPa (250 bar).

4.1.2 Rated charging pressure (RCP)

The RCP shall be specified by the manufacturer in order to prevent charging at a pressure that could result in the shell wall stress exceeding the design stress limit.

4.1.3 Stress level at MDP

The stress level at MDP shall be determined by the manufacturer from the hydrogen absorbing alloy's packing and expansion properties, the MDP within the MH assembly, and other applicable mechanical loadings.

4.2 Rated capacity

The manufacturer shall state the rated capacity of the MH assembly by units of mass of hydrogen.

4.3 Temperature ranges

4.3.1 Operating temperature range

The minimum and maximum temperature for normal operating conditions to which the MH assembly is rated shall be specified by the manufacturer.

4.3.2 Service temperature range

The minimum and maximum temperature for normal service conditions to which the MH assembly is rated shall be specified by the manufacturer. At a minimum, this range shall be of at least from −40 °C to 65 °C and shall include the entire operating temperature range.

4.4 Environmental conditions

The MH assemblies are expected to be exposed to a number of environmental conditions over their service life, such as vibration and shock, varying humidity levels, and corrosive environments. The manufacturer shall specify the environmental conditions for which the MH assembly was designed.

4.5 Service life

The service life for the MH assemblies shall be specified by the manufacturer on the basis of use under service conditions specified herein. The service life shall not exceed that specified by the standard to which the shell is designed as per 5.3.

4.6 Hydrogen quality

The minimum quality of the hydrogen gas that shall be used to fill an MH assembly shall be specified by the manufacturer according to ISO 14687 or as appropriate.

NOTE If the quality of the hydrogen gas is considered a critical issue to avoid performance degradation of the MH assembly, the manufacturer may consider including the information on the product label.

4.7 Special service conditions

Any additional service conditions that shall be met for the safe operation, handling and usage of the MH assembly shall be specified by the manufacturer.

5 Design considerations

5.1 General

The MH assembly shall be designed and constructed to prevent leakage of hydrogen or metal hydride particles under normal conditions of storage and transport.

5.2 Material selection

5.2.1 General

The MH assembly components shall be made of materials that are suitable for the range of conditions expected over the life of the MH assembly. Components that are in contact with gaseous hydrogen and/or metal hydride material shall be sufficiently resistant to their chemical and physical action under normal service conditions to maintain operational and pressure containment integrity.

Hydrogen absorbing alloys and/or metal hydride materials that are classified as Type I explosive materials according to the UN Recommendations on the Transport of Dangerous Goods shall not be used in an MH assembly.

5.2.2 External surfaces

The MH assembly shell, shut-off valve, PRDs and other appurtenances shall be resistant to the environmental conditions specified in 4.4. Resistance to these environmental conditions may be provided by using materials inherently resistant to the environment or by applying resistant coatings to the components. Exterior protection may be provided by using a surface finish giving adequate corrosion protection (e.g. metal sprayed on aluminium or anodizing) or a protective coating (e.g. organic coating or paint). If an exterior coating is part of the design, the coating shall be evaluated using the applicable test methods specified in Annex B. Any coatings applied to MH assemblies shall be such that the application process does not adversely affect the mechanical properties of the shell or performance and operation of other components. The coatings shall be designed to facilitate subsequent in-service inspection and the manufacturer shall provide guidance on coating treatment during such inspections to ensure the continued integrity of the MH assembly.

5.2.3 Compatibility

The compatibility of MH assembly materials with process fluids and solids, specifically embrittlement due to the exposure to hydrogen, shall be considered. Guidance on compatibility of materials with gases is given in ISO 11114-1 and ISO 11114-2. Materials necessary for the pressure containment and structural integrity of the MH assembly and its internal and external appurtenances shall be resistant to hydrogen embrittlement, hydrogen attack and reactivity with contained materials and maintain their integrity for the service life of the MH assembly. Recognized test methods, such as those specified in ISO 11114-4, shall be used to select metallic materials resistant to hydrogen embrittlement where required for pressure or structural integrity. Consideration shall be given to the impact that temperature may have on hydrogen embrittlement.

Consideration shall be given to all of the chemical species that may be present during the charged, partially charged and discharged states and their potential reactivity with the MH assembly material. The MH assembly materials shall be selected so as the combination does not endanger the MH assembly integrity.

NOTE The susceptibility to hydrogen embrittlement of some commonly used metals is summarized in ISO/TR 15916. Additional guidance regarding hydrogen compatibility is found in Annex A.

5.2.4 Temperature

The MH assembly materials shall be suitable for the service temperature range specified in 4.3.2.

5.3 Shell design

5.3.1 Shells with internal volume greater than 120 ml

The MH assembly shell shall be designed and tested according to ISO 7866, ISO 9809-1, ISO 9809-3, ISO 11119-1, ISO 11119-2, or standards registered in accordance with ISO 16528-1, as applicable. Shells designed and tested in accordance with ISO 9809-1 shall have a tensile strength less than 950 MPa. Shells designed and tested in accordance with ISO 11119-1 or ISO 11119-2 that use seamless steel liners conforming to ISO 9809-2 or to ISO 9809-1 shall have a tensile strength less than 950 MPa.

The shell shall not exceed 150 l internal volume, and the MDP shall not exceed 25 MPa (250 bar). The maximum combined stresses for the loads described in 5.4 as well as the operating and service temperature ranges for the MH assembly shall not exceed the limits prescribed by the standard to which the shell is designed.

NOTE 1 Shells can be designed and tested according to one of the International Standards specified above, even where the shell internal volume is less than that covered by the scope of that International Standard.

NOTE 2 An equivalent gas pressure calculated to be equal to the stress level at MDP can be used as the design hydraulic test pressure for determining minimum shell wall thickness.

5.3.2 Shells with internal volume of 120 ml or less

For MH assemblies with an internal volume of 120 ml or less, the shell design shall be deemed to be appropriate if the shell meets 5.3.1 or the MH assembly meets the following design and test criteria:

a) the pressure in the MH assembly shall not exceed 5 MPa (50 bar) at 55 °C when the MH assembly is filled to its rated capacity; and

b) the MH assembly design shall withstand as required by 6.2.3, without leaking or bursting, a minimum shell burst pressure of 2 times the pressure in the MH assembly at 55 °C when filled to rated capacity, or 1,6 times the pressure in the MH assembly at the maximum service temperature when filled to rated capacity, or 200 kPa (2 bar) more than the MDP of the assembly at 55 °C when filled to rated capacity, whichever is greater.

5.4 Design strength

The shell design shall take into account the stress level at 1,25 times MDP. Consideration of components contributing to the stress level at MDP shall include but not be limited to:

— 1,25 × MDP;

— thermal stress, including dissimilar rates of thermal expansion and contraction;

— weight of internals in any possible MH assembly orientation;

— shock and vibration loading;

— maximum stress due to hydrogen absorbing alloy expansion;

— other mechanical loadings.

To verify that the design stress limit is not exceeded, the MH assembly design shall be subjected to the hydrogen cycling and strain measurement test described in 6.2.6.

NOTE The process of introducing and subsequently removing hydrogen in the hydrogen absorbing alloy typically causes it to expand and contract. In turn, this can result in large stresses inside the alloy's particles that cause them to fragment into smaller particles, a phenomenon known as decrepitation. After several charge/discharge cycles, the average particle size may have significantly decreased. Stresses on the MH assembly walls may be derived from expansion of the hydrogen absorbing alloy during hydrogenation and from changes in the packing configuration due to decrepitation over the service life of the MH assembly. The magnitude of the expansion/contraction phenomena will vary greatly as a function of the hydrogen absorbing alloy used.

5.5 Overpressure and fire protection

5.5.1 General

The MH assembly shall be protected with one or more PRDs of the non-reclosing type, such as fusible triggers, rupture disks and diaphragms, or of the re-sealable type, such as spring-loaded PRVs. The MH assembly and any added component (e.g. insulation or protective material) shall collectively pass the fire test specified in 6.2.2. The PRD shall conform to the requirements of 5.5.2 and 5.5.3 and the additional requirements of the competent authority of country of use, as applicable.

For MH assemblies with an internal volume of 120 ml or less, other means may be used to protect from overpressurization, such as venting through a feature integral to the shell. MH assemblies that use an alternative means of relieving pressure shall meet the acceptance criteria of the fire test specified in 6.2.2.

5.5.2 PRD activation pressure

The pressure of actuation of pressure-activated PRDs shall be specified by the manufacturer and shall be greater than the MDP but less than 1,25 times the MDP. In no case shall the pressure of actuation of a pressure-activated PRD exceed the test pressure of the shell. For PRVs, the full flow capacity pressure shall also be specified, and shall not exceed the test pressure of the shell.

5.5.3 PRD activation temperature

The temperature at which any thermally actuated PRD is set to activate shall be specified by the manufacturer and correspond to an equilibrium pressure inside the MH assembly of less than 1,25 times the MDP. In no case shall the temperature of actuation of a temperature-activated PRD result in an equilibrium pressure inside the MH assembly that exceeds the test pressure of the shell. The PRD shall have a pressure rating greater than the MDP at all temperatures less than or equal to 10 °C above the maximum service temperature. In no case shall the PRD activate at a temperature lower than the maximum service temperature.

5.6 Loading of hydrogen absorbing alloy

Procedures and verification testing shall be put in place to ensure the consistent loading of the hydrogen absorbing alloy/metal hydride in the MH assembly.

5.7 Shut-off valves

5.7.1 General

The MH assembly shall incorporate a shut-off valve that shall be capable of being closed when the MH assembly is disconnected from the refill or gas-consuming equipment. The shut-off valve may be manually actuated, such as by a handwheel, or automatically actuated.

All MH assemblies shall provide a means of shut-off valve protection that complies with 5.7.4 or 5.7.5.

The shut-off valve selection shall include verification that the shut-off valve seal is maintained with vacuum conditions within the MH assembly.

NOTE Due to the temperature/pressure characteristics of metal hydrides, the development of sub-ambient pressures are possible within MH assemblies.

5.7.2 MH assemblies with internal volume greater than 120 ml

Shut-off valves shall comply with ISO 10297:2006, or equivalent, with the following exceptions:

a) 3 times MDP shall be used as the test pressure;

b) Valve test pressure, p_{vt}, shall be equal to 1,5 times the MDP.

In addition, the shut-off valve shall meet all requirements and tests prescribed in this International Standard.

Alternatively, if the shut-off valve cannot demonstrate full compliance to ISO 10297:2006 or equivalent, the shut-off valve construction and performance shall meet all the requirements and tests prescribed in this International Standard as well as the following requirements:

— the material requirements of ISO 10297:2006, 4.3;

— the test requirements of ISO 10297:2006, 6.1 to 6.8, as they apply to the tests prescribed below with the exception the valve test pressure, p_{vt}, shall be equal to 1,5 times the MDP;

— the hydraulic pressure test of ISO 10297:2006, 6.9, with the exception that 3 times the MDP shall be used as the test pressure;

— the leak tightness test of ISO 10297:2006, 6.11, where p_{vt} shall be equal to 1,5 times the MDP;

— the endurance test of ISO 10297:2006, 6.12, using a gas pressure equal to 0,5 times the MDP. When the shut-off valve does not incorporate a handwheel, the forces and torques used in the endurance test shall be representative of those used in service to open and close the valve member. Prior to and following the endurance test, the shut-off valve shall be tested for leakage from an internal and external leakage perspective at a test pressure of 1,5 times MDP at minimum and maximum service temperature. Leakage rates less than or equal to 6 standard cm^3/h (standard conditions of 0 °C and 101,325 kPa absolute) shall be acceptable.

The minimum rated pressure of the shut-off valve shall be at least equal to 1,5 times MDP.

In addition, the shut-off valve manufacturer shall demonstrate that the shut-off valve is subjected to the requirements of ISO 14246.

5.7.3 MH assemblies with internal volume of 120 ml or less

For MH assemblies with an internal volume of 120 ml or less, the shut-off valve construction and performance shall meet all requirements and tests prescribed in this International Standard as well as the following requirements:

— the material requirements of ISO 10297:2006, 4.3;

— the test requirements of ISO 10297:2006, 6.1 to 6.8, as they apply to the tests prescribed below with the exception that the valve test pressure, p_{vt}, shall be equal to 1,5 times the MDP;

— the hydraulic pressure test of ISO 10297:2006, 6.9, with the exception that the test pressure shall be in accordance with 5.3.2 b) and the test may be performed pneumatically;

— the leak tightness test of ISO 10297:2006, 6.11, where p_{vt} shall be equal to 1,5 times the MDP. Valve closure may be determined using torque, compression or other suitable means and the test gas shall be helium;

— the endurance test of ISO 10297:2006, 6.12, using a gas pressure equal to 0,5 times the MDP and maximum number of 100 cycles. When the shut-off valve does not incorporate a handwheel, the forces and torques used in the endurance test shall be representative of those used in service to open and close the valve member. Prior to and following the endurance test, a shut-off valve shall be tested for leakage from an internal and external leakage perspective at a test pressure of 1,5 times MDP at minimum and maximum service temperature. Leakage rates less than or equal to 3 standard cm^3/h (standard conditions of 0 °C and 101,325 kPa absolute) shall be acceptable.

The minimum rated pressure of the shut-off valve shall be at least equal to the MDP.

In addition, the shut-off valve manufacturer shall demonstrate that the shut-off valve is subjected to the requirements of ISO 14246.

5.7.4 Integral shut-off valve protection

A MH assembly design that uses an integral method of shut-off valve protection that is not meant to be removed for MH assembly operation, such as the use of a shroud, collar or recessing the valve in the MH assembly, shall meet the requirements of the drop test in 6.2.4.

5.7.5 Removable shut-off valve protection

MH assembly designs that use a removable means of shut-off valve protection that is meant to be removed for MH assembly operation, such as a cover, cap or guard, shall meet the requirements of the drop test in 6.2.4 with the protective means in place and meet the requirements of the shut-off valve impact test in 6.2.7 without the protective means in place.

Removable means of shut-off valve protection, having passed the drop test in 6.2.4, shall be acceptable for use only with filled MH assemblies at a mass equal to or less than the mass tested and with MH assemblies with shut-off valves of dimensions not exceeding those of the tested shut-off valve.

5.8 Actively cooled MH assemblies

MH assemblies that employ an active cooling system to control and/or affect system temperature shall be designed to ensure that there will be no inadvertent leakage of fluid between the MH assembly and the cooling system. The cooling system shall be employed when performing the hydrogen cycling and strain measurement test in 6.2.6.

5.9 Particulate containment

Particulate matter shall not impede the functioning of the valves or PRDs. A means of particulate matter containment may be used to achieve this purpose. The MH assemblies shall meet the requirements of the fire test of 6.2.2 and the hydrogen cycling and strain measurement test of 6.2.6.

6 Inspection and testing

6.1 General

Evaluation of conformity shall be performed in accordance with the relevant regulations of the country where the MH assemblies are used.

In order to ensure that the MH assemblies are in compliance with this International Standard, they shall be subject to inspection and testing in accordance with this clause. Inspection and testing shall be performed by an authorized inspection body recognized in the countries of use.

NOTE Some countries do not require inspection and testing to be carried out by an authorized inspection body.

6.2 Type/qualification tests

6.2.1 General

The following type tests shall be performed to qualify an MH assembly design. The MH assemblies used for the type tests shall be representative of production MH assemblies. The data for all type tests shall be acquired using calibrated instruments.

Any change in shell design, hydrogen absorbing alloy, manufacturing process or loading procedure of hydrogen absorbing alloy shall require repeating the fire test of 6.2.2, the drop test of 6.2.4 and the hydrogen cycling and strain measurement test of 6.2.6, and, if applicable, the thermal cycling test of 6.2.8.

Compliance to this International Standard shall be recorded for each MH assembly design on a type approval certificate. An example of a suitably worded certificate is given in Annex C.

6.2.2 Fire test

6.2.2.1 General

The fire test shall be performed on all new MH assembly designs to demonstrate that the fire protection system, such as PRD and/or integral thermal insulation, will prevent the rupture of the MH assembly under the specified fire conditions.

Any significant change to the design as defined in the standard (see 5.3.1) to which the shell is designed (including, but not limited to, changes in diameter, length, shell material type and minimum design thickness) and any change to the type, number or flow capacity of the PRD, means of solid particulate containment or in the hydrogen absorbing alloy shall necessitate repeating the fire test.

As an exception, a manufacturer may use data and engineering calculations, based on previous fire test results on existing designs, in cases involving design changes that would reduce the risk of shell failure in the fire test (e.g. reduction in shell length, or increase in PRD flow capacity), to show that a new design does not require repeating the fire test.

Precautions should be taken to ensure safety of personnel and property during the fire test in the event that an MH assembly rupture occurs.

6.2.2.2 Sample preparation

The MH assembly shall be filled to rated capacity with hydrogen.

6.2.2.3 Data monitoring and recording

The temperature and pressure of the MH assembly shall be monitored remotely and recorded at intervals of ⩽15 s. A valve shall be installed to allow venting of the MH assembly in the event of a malfunction of the test equipment or system.

In addition to the temperature and pressure readings, the following information shall also be recorded for each test:

— MH assembly manufacturer;

— MH assembly part or model number;

— unique identifier;

— PRD-type and rating;

— PRD location and orientation;

— date of test;

— MH assembly RCP;

— number of charge/discharge cycles that the MH assembly has undergone;

— MH assembly orientation (vertical, horizontal or inverted);

— ambient temperature;

— estimated wind condition/direction;

— names of witnesses;

— time of activation of PRD; and

— elapsed time to completion of the test.

For MH assembly designs that contain small quantities of hydrogen that preclude accurate monitoring of pressure during the fire test, a statement of justification for not monitoring the pressure during the fire test shall be provided, along with a description of the means for determining activation of the PRD.

6.2.2.4 Test set-up, fire source and test method

The fire tests shall be conducted on at least three MH assemblies in each orientation of intended use and/or transportation. For MH assembly designs for which the orientation of use and transportation are not specified, at least three MH assemblies shall be fire tested in each of the vertical and horizontal orientation and any other orientation due to asymmetry of the MH assembly design, if applicable. The tests shall include at least one test with the PRD oriented towards the fire source and at least one test with the PRD oriented 180° away from the fire source.

The MH assemblies, over their entire width, shall be subjected to a fire source of a maximum length of 1,65 m. For MH assemblies less than 1,65 m in length, the fire source shall totally engulf the MH assembly. MH assemblies longer than 1,65 m or equipped with multiple PRDs with a spacing greater than 1,65 m, shall be subjected to a partial engulfment fire test in the horizontal orientation. If an MH assembly is longer than 1,65 m and is fitted with a PRD at one end, the opposite end of the MH assembly shall be subjected to the fire source. If the MH assembly is fitted with PRDs at both ends, or at more than one location along the length of the MH assembly, the fire source shall be centred midway between the PRDs that are separated by the greatest horizontal distance.

For MH assemblies less than or equal to 0,30 m in length, a temperature-indicating device shall be installed within 0,05 m of, but not in contact with, the MH assembly surface near each end. For MH assemblies greater than 0,30 m in length, a temperature-indicating device shall be installed at each end and one at the midpoint. Temperature-indicating devices may be inserted into small metallic blocks (less than 0,025 m per side).

MH assemblies shall be subjected to a direct flame impingement test. Sufficient fuel shall be supplied to ensure a burn time of at least 20 min. The MH assembly shall be placed in the test orientation with the MH assembly at least 0,1 m above the fuel or at a greater height to ensure total flame engulfment. The fire shall produce a flame that totally engulfs the MH assembly. Shielding shall be used to prevent direct flame impingement on the shut-off valve, fittings, and/or PRD(s). The shielding shall not be in direct contact with the specified fire protection system.

Any fuel may be used for the fire source, provided it supplies uniform heat sufficient to maintain the specified test conditions for a minimum of 20 min. The fire test should to be carried out in a properly ventilated facility or in open ground for safety. The selection of a fuel should take into consideration air pollution concerns. The arrangement of the fire shall be recorded in detail to ensure that the rate of heat input to the MH assembly is reproducible.

NOTE MH assemblies that have been subjected to the cycling and strain measurement test of 6.2.6 may be used in this test.

6.2.2.5 Acceptance criteria

Any failure or inconsistency of the fire source during a test shall invalidate the result, and a re-test shall be carried out. Any venting through, or rupture of, the shell, valve, fitting or tubing during the test that is not part of the intended protection system, shall invalidate the result and a re-test shall be carried out.

The MH assembly design shall be deemed to have passed the fire test if, for all valid tests, there is no generation of projectiles and one of the following criteria is met:

— the PRD or other venting method of all MH assemblies subjected to the fire test vent each MH assembly to zero gauge pressure without rupture of the MH assembly; or

— all MH assemblies subjected to the fire test withstand the fire for a minimum of 20 min without rupture.

6.2.3 Initial burst tests for MH assemblies with an internal volume of 120 ml or less

At least three MH assemblies shall be subjected to an initial burst test to demonstrate compliance to 5.3.2 b). Either a hydrostatic or a pneumatic burst test shall be performed; however, all tests shall be performed in the same manner. All bursts shall occur in the same manner for all tests performed.

Adequate precautions should be taken to ensure safety of equipment and personnel. In particular, during a pneumatic burst testing, personnel should be aware of the potential for releases of large amounts of stored energy and potentially hazardous materials as a result of the burst.

6.2.4 Drop test

6.2.4.1 General requirements

All MH assembly designs shall meet the requirements of the drop test. Any significant change to the design as defined in the standard (see 5.3.1) to which the shell is designed (including, but not limited to, changes in diameter, length, shell material type and minimum design thickness) and any change in shut-off valve, means of solid particulate containment or loaded mass of the hydrogen absorbing alloy shall necessitate repeating the drop test.

The surface onto which the MH assemblies are dropped shall be a smooth, horizontal, concrete or steel surface. The container shall be allowed to bounce on the concrete or steel surface after the initial impact. No attempt shall be made to prevent this secondary impact. A guide rail for posture maintenance may be used, provided that it does not reduce the free-fall velocity.

6.2.4.2 Sample preparation

The MH assemblies used for these tests shall include their integral or removable shut-off valve protection (see 5.7.4 and 5.7.5). The MH assemblies shall have an equivalent weight (± 2 %), packing density and internal structure as production MH assemblies. Ballast material may be used in place of the hydrogen absorbing alloy. The MH assemblies shall not be pressurized.

6.2.4.3 Test procedure

MH assemblies shall be drop tested in accordance with the following conditions. One MH assembly may be used for all drop tests performed in a) to c). The drop tests shall be carried out at room temperature (20^{+10}_{-5}) °C.

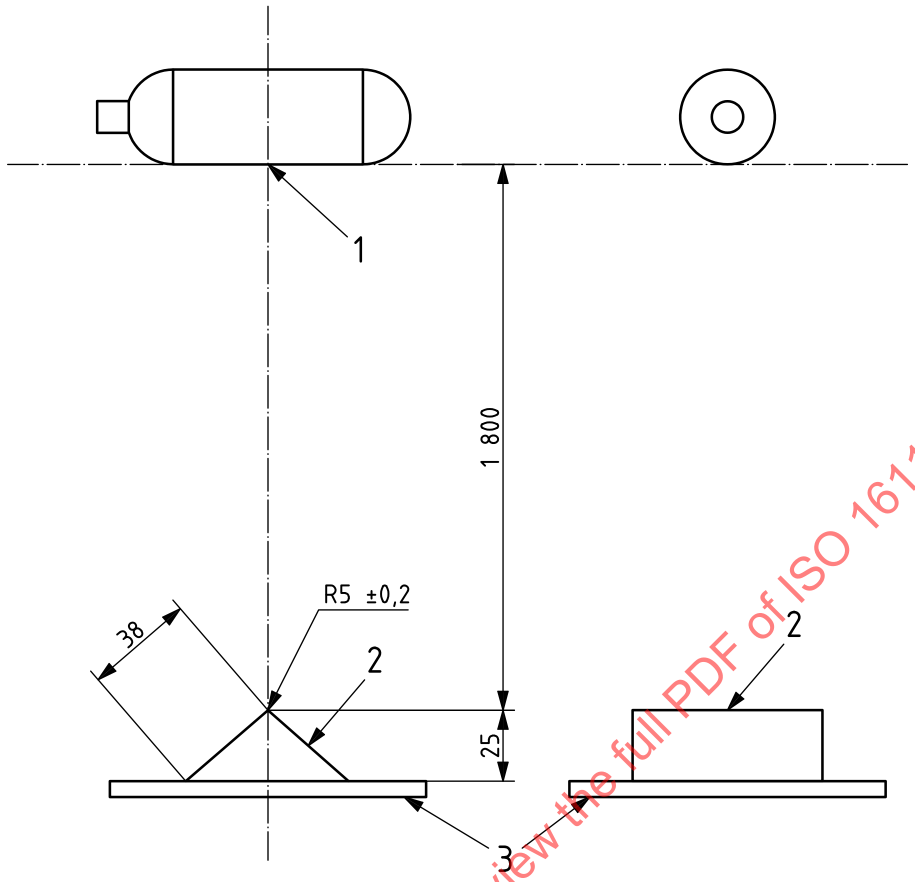
a) One MH assembly shall be dropped vertically on the end containing the shut-off valve assembly. One MH assembly shall be dropped vertically on the end opposite the shut-off valve assembly. In both cases, the MH assembly shall be dropped from a height of not less than 1,8 m measured from the lower end of the MH assembly.

b) One MH assembly shall be dropped at a 45° angle on the end containing the shut-off valve assembly from a height such that the centre of gravity is at a minimum height of 1,8 m. If the lower end of the MH assembly is at a height of less than 0,6 m, the drop angle shall be changed to maintain the lower end of the MH assembly and the centre of gravity at a minimum height of 0,6 m and 1,8 m respectively. When the shut-off valve, PRD and other appurtenances are set on both ends of the MH assembly, the MH assembly shall be dropped at a 45° angle on its weakest end.

c) One MH assembly shall be dropped horizontally from a height of 1,8 m onto a steel apex as shown in Figure 1. The MH assembly shall be placed such that its centre of gravity is aligned with the rounded edge of the steel apex as shown in Figure 1. In order to prevent movement of the steel apex by the collision of the MH assembly, the steel apex shall be fixed to the concrete pad or flooring. The MH assembly shall strike the steel apex before striking the concrete pad or flooring.

d) For shells of composite design (such as shells designed according to ISO 11119-1 or ISO 11119-2) at least one additional MH assembly shall be dropped according to a) and b).

Dimensions in millimetres



Key

1 centre of gravity

2 steel apex

3 smooth, horizontal concrete pad or flooring

Figure 1 — MH assembly drop test onto an apex

6.2.4.4 Acceptance criteria

6.2.4.4.1 General

The shut-off valve shall remain operational (i.e. capable of being opened and closed) after all drop tests.

All MH assemblies that have undergone the drop tests shall be visually inspected and all apparent damage recorded. All MH assemblies shall be subjected to the leak test of 6.2.5 at a temperature of (20^{+10}_{-5}) °C and MDP and meet the acceptance criteria.

MH assemblies dropped in accordance to 6.2.4.3 d), shall additionally be subjected to the ambient cycle test of ISO 11119-2:2002, 8.5.5, and withstand 3 000 pressurization cycles at five-sixths of the MDP without failure by burst or leakage.

After successful completion of the leak test and, if applicable, the ambient cycle test specified above, all MH assemblies shall be pressurized to destruction as per 6.2.4.4.2 or 6.2.4.4.3 and meet the acceptance criteria.

6.2.4.4.2 MH assemblies with internal volume greater than 120 ml

The MH assemblies shall be pressurized to destruction using a hydrostatic burst test. The recorded burst pressures shall exceed 85 % of the minimum shell burst pressure specified by the standard to which the shell was designed. All bursts shall occur in a manner consistent with the standard to which the shell was designed and in the same manner for all tests performed.

6.2.4.4.3 MH assemblies with internal volume of 120 ml or less

The MH assemblies shall be pressurized to destruction using a hydrostatic or a pneumatic burst test. The recorded burst pressures shall exceed 85 % of the minimum shell burst pressure specified in 5.3.2. All bursts shall occur in a manner consistent with the initial burst test of 6.2.3 and in the same manner for all tests performed.

Adequate precautions should be taken to ensure safety of equipment and personnel. In particular, during a pneumatic burst testing, personnel should be aware of the potential for releases of large amounts of stored energy and potentially hazardous materials as a result of the burst.

6.2.5 Leak test

6.2.5.1 Test procedure

The MH assembly shall be charged with hydrogen, helium, or a blend of the two, and monitored for leaks at the conditions indicated in Table 1.

Table 1 — Temperature/pressure conditions for leak test

Temperature	Pressure
Minimum service temperature	RCP
(20^{+10}_{-5}) °C	RCP
Maximum service temperature	MDP

NOTE Before placing the MH assembly in an enclosed area to perform the leak test of either 6.2.5.2.1 or 6.2.5.2.2, it is recommended to test for the presence of major leaks using a soap bubble solution, or by other adequate means, on all possible leak locations.

6.2.5.2 Acceptance criteria

6.2.5.2.1 MH assemblies with internal volume greater than 120 ml

The total hydrogen leak rate shall be less than 6 standard cm^3/h (standard conditions of 0 °C and 101,325 kPa absolute). If hydrogen gas is not used, the leak rate shall be converted into an equivalent hydrogen leak rate.

6.2.5.2.2 MH assemblies with internal volume of 120 ml or less

The total hydrogen leak rate shall be less than 3 standard cm^3/h (standard conditions of 0 °C and 101,325 kPa absolute). If hydrogen gas is not used, the leak rate shall be converted into an equivalent hydrogen leak rate.

6.2.6 Hydrogen cycling and strain measurement test

6.2.6.1 General

The hydrogen cycling and strain measurement test shall be performed on all new MH assembly designs to demonstrate that the design stress limits of the shell are not exceeded during use. Any significant change to the design as defined in the standard (see 5.3.1) to which the shell is designed (including, but not limited to, changes in diameter, length, shell material type and minimum design thickness) and means of solid particulate containment or formulation of or loaded mass of the hydrogen absorbing alloy shall necessitate repeating the hydrogen cycling and strain measurement test. MH assemblies that employ an active cooling system to control and/or affect system temperature shall be subjected to the test with the cooling system in place.

Precautions should be taken to ensure safety of personnel and property during testing in the event that an MH assembly failure or hydrogen release occurs.

6.2.6.2 Test set-up

Each MH assembly shall be adequately instrumented with strain gauges to determine the maximum local strain that the shell experiences during cycling. With MH assemblies, the strain may not be uniform throughout the MH assembly. The number and location of the strain gauges required to measure the highest strain experienced by the shell may be determined from engineering models based on knowledge of the design, including stress distribution and analysis information provided by the shell manufacturer, the internal configuration and geometry, hydrogen absorbing alloy distribution, etc. If engineering models cannot accurately determine the points of expected highest strain, the number and locations of required strain gauges shall be empirically determined by extensively instrumenting at least two MH assemblies with strain gauges and performing the test. Based on the results, further testing may be performed using fewer strain gauges that are strategically placed to measure the highest strain levels experienced by the shell.

As a minimum, the hoop strain shall be monitored on cylindrical and dome sections of MH assemblies, bending strain shall be monitored on flat sections of MH assemblies and for strain concentration points (such as corners and edges), the strain in areas around the concentration point shall be monitored, and a concentration factor shall be used to estimate the strain at the concentration point.

The strain gauges shall be protected from damage during extended testing and exposure to the cycling environment, for example by the use of a chemically-resistant epoxy. Periodically during and, at least, at the start and end of cycling, the strain gauges shall be calibrated to ensure proper functioning. If any strain gauge is found to not be properly functioning, it shall be replaced.

The strain at the design stress limit shall be determined either by engineering calculations based on the shell design and material properties, or empirically by internally applying either a pneumatic or hydrostatic pressure up to a pressure equivalent to the shell design stress limit and measuring the strain. For any MH assembly where the strain gauges are applied to an outer layer and not directly to the shell or liner in contact with the metal hydride and hydrogen gas (such as shells of type II and III fibre-wrapped composite cylinder design) or for any shell that has been intentionally subjected to plastic deformation (i.e. autofrettage), the strain at the design stress limit for each gauge shall be determined empirically prior to cycling the MH assemblies with hydrogen.

6.2.6.3 Test method

For MH assemblies designed to be transported and used in a single orientation, at least five MH assemblies shall be tested in that orientation. For MH assembly designs that do not preclude use in more than one orientation, at least three MH assemblies shall be tested in two orientations perpendicular to each other, with the MH assembly axis horizontal and vertical. The MH assemblies shall be hydrogen charge cycled from not more than 5 % of rated capacity to not less than 95 % of rated capacity. The RCP shall be used for charging and the temperatures shall be held within the operating temperature range. The cycling shall be continued for at least 106 cycles and until the acceptable results defined in 6.2.6.4 are met. If the measured strain on consecutive cycles exceeds the design stress limit or plastic deformation of the shell material occurs, the testing shall be discontinued.

As a minimum, a measurement from each strain gauge shall be recorded on every cycle while at the maximum charge condition.

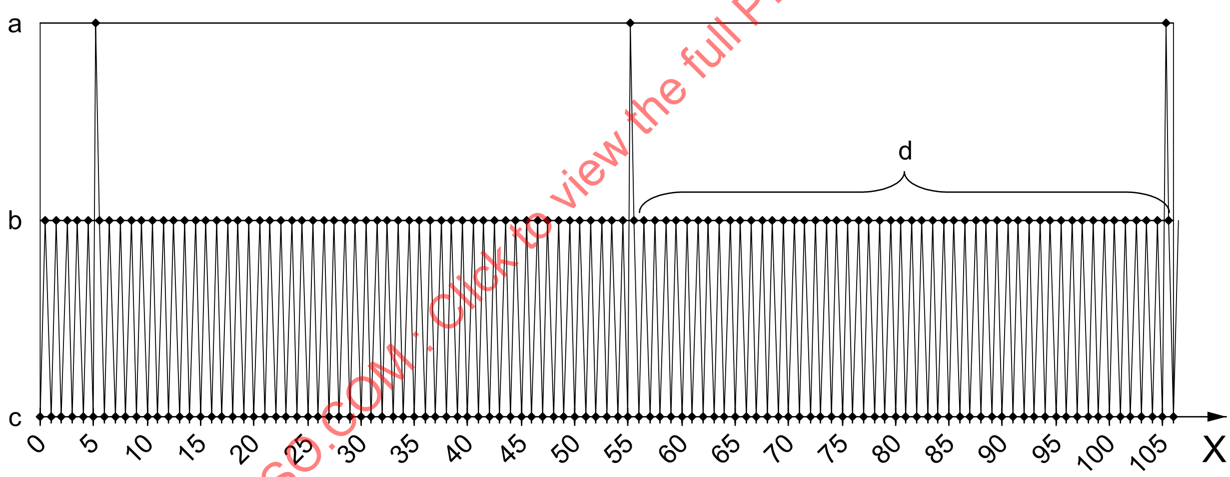
After the fifth complete cycle and then at intervals of not more than 50 cycles, with the MH assemblies charged to not more than 5 % of their rated capacity, each MH assembly shall be subjected to the following vibrational sequence while in the orientation for cycling:

— A sinusoidal waveform with a logarithmic sweep between 7 Hz and 200 Hz and back to 7 Hz traversed in 15 min. This cycle shall be repeated 12 times for a total of 3 h for each MH assembly. The logarithmic frequency sweep shall be as follows: from 7 Hz a peak acceleration of 1 g_n shall be maintained until 18 Hz is reached. The amplitude shall then be maintained at 0,8 mm (1,6 mm total excursion) and the frequency increased until a peak acceleration of 8 g_n occurs (approximately at 50 Hz). A peak acceleration of 8 g_n shall then be maintained until the frequency is increased to 200 Hz.

For MH assemblies with a mass greater than 100 kg, the following vibration sequence may be used as an alternative to the above sequence.

— Simple harmonic motion with a vertical amplitude of 0,8 mm with a 1,6 mm maximum total excursion. The frequency shall be varied at a rate of 1 Hz/min between the limits of 10 Hz to 55 Hz. The entire range of frequencies and return shall be traversed in (95 ± 5) min.

Figure 2 shows the minimum cycling requirements.



Key

X cycle number

Linear regression analysis shall show no increase in strain for the last 50 consecutive cycles (d); the cycling pattern is continued until this condition is met.

a vibrate
b charge
c discharge
d last 50 consecutive cycles

Figure 2 — Graphical depiction of minimum cycle requirements

6.2.6.4 Acceptance criteria

For each strain gauge in a period of 50 consecutive cycles, either the maximum measured strain shall not be greater than 50 % of the strain at the design stress limit, or, there is no trend of increasing strain. The MH assembly shall be considered to have failed the test and a redesign shall be required if, for any strain gauge, the strain for consecutive cycles exceeds the strain for the shell at the design stress limit or if the shell experiences plastic deformation.

To determine that there is no trend of increase in strain, the data for each strain gauge with a maximum strain greater than 50 % of the strain at the design stress limit shall be analysed by the least squares linear regression method, according to the equation:

$$a = \frac{\left(\sum_{i=j}^{j+N} y_i x_i\right) - N\,\overline{y}\,\overline{x}}{\left(\sum_{i=j}^{j+N} x_i^2\right) - N\,\overline{x}^{\,2}}$$

where

a is the coefficient indicating the slope of the measured strain data;

x is the cycle number;

$\overline{x} = \frac{1}{N}\sum_{i=j}^{j+N} x_i$ (average cycle number);

N shall be 50, the number of consecutive cycles analysed;

y the measured strain; and

$\overline{y} = \frac{1}{N}\sum_{i=j}^{j+N} y_i$ (the average strain).

The MH assembly shall be cycled until, for a period of 50 consecutive cycles, the coefficient a is less than or equal to zero for all strain gauges that have a strain reading greater than 50 % of the strain at the design stress limit. The 50 cycles analysed shall be the final 50 consecutive cycles performed. This criterion shall be met by all strain gauges on an MH assembly for the same period of consecutive cycles.

Additionally, after completion of the cycling and strain measurement test, all MH assemblies shall be pressurized, and with a blank-off on the outlet, the valve shall be cycled between the open and closed positions a minimum of two times. With the blank-off removed from the outlet, all MH assemblies shall meet the acceptance criteria of the leak test of 6.2.5. At least one MH assembly from each orientation tested shall be subjected to the fire test of 6.2.2 and meet the acceptance criteria.

Further, for MH assemblies with an internal volume of 120 ml or less, after completion of the leak testing, at least one MH assembly from each orientation tested shall be pressurized to destruction. The burst test may be either a hydrostatic or a pneumatic burst test. The recorded burst pressures shall exceed the minimum shell burst pressure specified in 5.3.2. All bursts shall occur in a manner consistent with the initial burst specified in 6.2.3 and in the same manner for all tests performed.

Adequate precautions should be taken to ensure safety of equipment and personnel. In particular, during pneumatic burst testing, personnel should be aware of the potential for releases of large amounts of stored energy and potentially hazardous materials as a result of the burst.

For MH assemblies that employ an active cooling system to control and/or affect system temperature, any inadvertent leakage between the MH assembly and cooling fluid shall be considered a failure to meet the acceptance criteria of this test.

6.2.7 Shut-off valve impact test

6.2.7.1 General

As indicated in 5.7.5, MH assembly designs that employ a removable means of valve protection shall be subjected to the following shut-off valve impact test.

6.2.7.2 Sample preparation

Three MH assemblies shall be subjected to this shut-off valve impact test. For the purpose of this test, ballast may be used in place of the hydrogen absorbing alloy or the shell may be left empty. The MH assemblies shall not be pressurized with gas during the test. The removable shut-off valve protection shall be removed for this test.

6.2.7.3 Test procedure

A hardened steel ball or an impact object tipped with a hardened steel ball shall be used for this test. The hardened steel ball shall have a Brinell hardness of 248 ± 3 and its diameter shall be allowed to vary with respect to the size of the shut-off valve to allow it to strike the side of the valve 90° to the longitudinal axis of the valve and co-incident with a plane passing through the same axis.

The hardened steel ball, or the impact object tipped with a hardened steel ball, as well as the MH assembly, shall be conditioned for at least 4 h at −40 °C. Within 5 min after conditioning, the MH assembly shall be rigidly anchored and the shut-off valve shall be subjected to the following two impacts. The first impact shall strike the side of the shut-off valve 90° to the longitudinal axis of the valve and co-incident with a plane passing through the same axis. The points of impact on the shut-off valve shall not be obstructed by features such as outlet connecting threads, pressure relief devices, handwheel, etc. The hardened steel ball or the impact with a hardened steel ball object shall have sufficient mass and velocity to impart the minimum energy specified in Table 2. After the first impact, the MH assembly shall be rotated 180° and a second side impact test shall be conducted on the other side of the shut-off valve.

Table 2 — Ball impact requirements for valves

MH assembly type (V = internal volume in litres)	**Minimum energy (E)** [a] joules
$V \leqslant 0{,}35$	1,02
$0{,}35 < V \leqslant 10$	6,80
$10 < V \leqslant 25$	13,50
$25 < V \leqslant 100$	27,10
$100 < V$	162,70

[a] For example, for a free falling impact object tipped with a hardened steel ball,

$$E = mg_c h$$

where

E is energy, expressed in joules (J);

m is mass of the impact object tipped with a hardened steel ball, expressed in kilograms (kg);

g_c is the acceleration due to gravity (9,8 m/s²);

h is the vertical drop height, expressed in metres (m).

6.2.7.4 Acceptance criteria

Following the two impacts tests, each shut-off valve and MH assembly shall be visually inspected for damage and subjected to the leak test of 6.2.5 at (20^{+10}_{-5}) °C and MDP and meet the requirements therein.

The shut-off valve connection (inlet threads) shall remain intact without cracking and the shut-off valve shall be operative. A break of the handwheel shall not be considered as a failure to meet the test requirements as long as the shut-off valve is still capable of being opened and closed.

If the requirements of the leak test are not met or the shut-off valve does not remain operational after the tests, the test shall be repeated on three MH assemblies fitted with their removable shut-off valve protection. If the three MH assemblies meet the acceptance criteria, the design shall be considered as acceptable, provided each MH assembly is marked in accordance with 7.2.3.

6.2.8 Thermal cycling test

6.2.8.1 General

The thermal cycling test shall be performed on MH assemblies with an internal volume of 120 ml or less only.

NOTE This test is being performed to address potential concerns regarding not having performed a pressure cycling test similar to that prescribed in the ISO cylinder standards. This test is intended to thermally cycle a complete MH assembly over its service temperature range.

6.2.8.2 Test set-up

For MH assemblies designed to be transported and used in a single orientation, at least five MH assemblies shall be tested in that orientation. For MH assembly designs that do not preclude use in more than one orientation, at least three MH assemblies shall be tested in two orientations perpendicular to each other, with the MH assembly axis horizontal and vertical.

The MH assembly shall be filled to rated capacity with hydrogen. The MH assembly shall be placed in a temperature-controlled test chamber capable of cycling from minimum service temperature to maximum service temperature and vice versa over a period of 2 h.

6.2.8.3 Test procedure

The MH assemblies shall be subjected to the following thermal cycles (see Figure 3).

a) Place the MH assembly in the temperature-controlled test chamber and increase the chamber temperature from (20^{+10}_{-5}) °C to the maximum service temperature with a tolerance of ± 5 °C in 1 h ± 5 min.

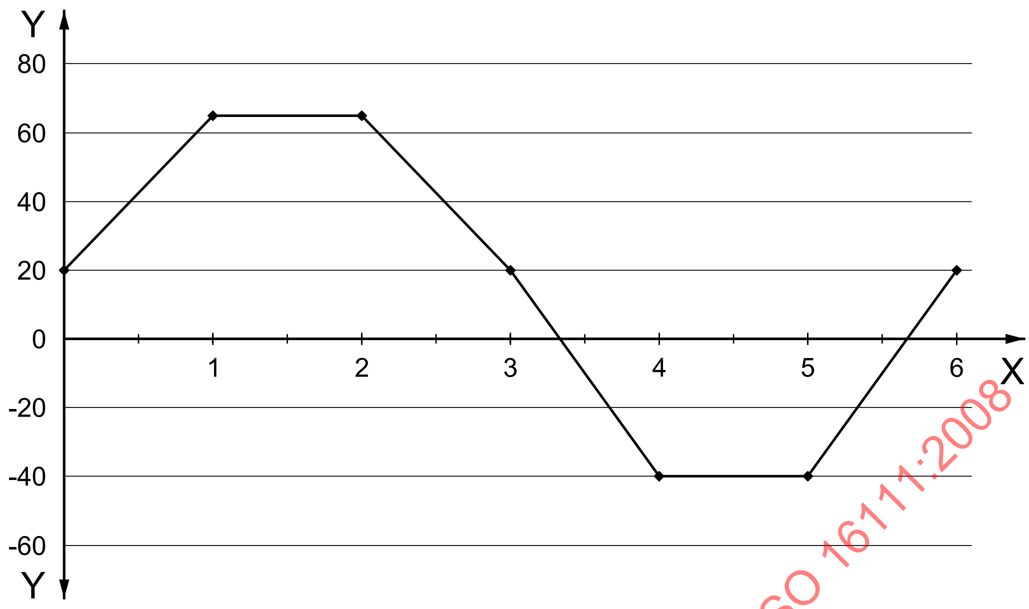
b) Keep the MH assembly at the maximum service temperature with a tolerance of ± 5 °C for a minimum of 1 h.

c) Decrease the chamber temperature to (20^{+10}_{-5}) °C in 1 h ± 5 min, then decrease the chamber temperature to the minimum service temperature with a tolerance of ± 5 °C in 1 h ± 5 min.

d) Hold the chamber temperature at the minimum service temperature with a tolerance of ± 5 °C for a minimum of 1 h.

e) Increase the chamber temperature to (20^{+10}_{-5}) °C in 1 h ± 5 min.

f) Repeat steps a) to e) 50 times.



Key

X time (h)

Y temperature (°C)

Figure 3 — Temperature cycling test example cycle

6.2.8.4 Acceptance criteria

Each MH assembly shall be subjected to, and meet the acceptance criteria of, the leak test of 6.2.5 following the thermal cycling test.

After completion of the leak testing, at least one MH assembly from each orientation tested shall be pressurized to destruction. The burst test may be either a hydrostatic or a pneumatic burst test. The recorded burst pressures shall exceed the minimum shell burst pressure specified in 5.3.2. All bursts shall occur in a manner consistent with the initial burst test specified in 6.2.3 and in the same manner for all tests performed.

Adequate precautions should be taken to ensure safety of equipment and personnel. In particular, during pneumatic burst testing, personnel should be aware of the potential for releases of large amounts of stored energy and potentially hazardous materials as a result of the burst.

6.2.9 Type test reports

The type test reports verifying compliance with the requirements of this International Standard shall be made available to users upon request.

6.3 Batch tests

6.3.1 General requirements

Batch testing shall be conducted at specified intervals during manufacturing to ensure consistency of the manufactured MH assemblies with the prototype design.

The size of a batch shall be determined by the manufacturer with consideration to the volume of MH assembly and the material of construction. A batch shall consist of no more than one lot of hydrogen absorbing alloy and not greater than the batch size of the shell as defined in the shell standard, or as approved by the competent authority.

All batch tests of the MH assembly shall be carried out on finished MH assemblies.

6.3.2 Burst test

At least one MH assembly from each batch shall be pressurized to destruction.

For MH assemblies with internal volume greater than 120 ml, the burst tests methods and acceptance criteria shall meet the requirements of the standard (see 5.3.1) to which the shell is designed.

For MH assemblies with an internal volume of 120 ml or less, the burst tests shall be performed in accordance with 6.2.3. All bursts shall occur in a manner consistent with the initial bursts tests specified in 6.2.3 and in the same manner for all tests performed.

Adequate precautions should be taken to ensure safety of equipment and personnel. In particular, during pneumatic burst testing, personnel should be aware of the potential for releases of large amounts of stored energy and potentially hazardous materials as a result of the burst.

6.4 Routine tests and inspections

6.4.1 Routine tests

The manufacturer shall perform routine tests and inspection on each MH assembly and maintain records for not less than 20 years or 1,5 times the service life of the MH assembly, whichever is longer.

As part of the routine tests, each completed MH assembly shall be subjected to the leak test of 6.2.5 at (20^{+10}_{-5}) °C and RCP and meet the acceptance criteria.

For all shells used in the manufacturing of MH assemblies, the MH assembly manufacturer shall obtain and maintain the documentation verifying that the shell was manufactured, tested and qualified in accordance to the shell standard. The MH assembly manufacturer shall also perform incoming inspection of shells to the degree necessary to ensure that the shells meet the specified requirements.

6.4.2 Certificates of manufacture

A certificate of manufacture shall be prepared for each batch of MH assemblies that meets the requirements of this International Standard in all respects. An example of a suitably worded certificate is given in Annex D.

7 Marking, labelling, and documentation

7.1 Marking

The MH assembly shall have, as a minimum, the following information permanently marked in a clearly visible location:

a) a reference to this International Standard;

b) the RCP in bar;

c) the manufacturer's identification;

d) the date of manufacturing (month and year);

e) a manufacturer's serial or unique identification number; and

f) the date of expiry based on the maximum service life (month and year).

In cases where, due to size or area limitations, it is not possible to include all of the above information in a legible format, the use of a traceable code may be used. If a traceable code is used, the MH assembly shall still be permanently marked with the RCP, the manufacturer's serial or unique identification number and the date of expiry as per b), e) and f).

7.2 Labelling

7.2.1 General

The precautionary labelling shall be in accordance with ISO 7225. Labels shall not obscure any permanent shell markings.

In cases where, due to size or area limitations, it is not possible to include all information on the label, the information may be included on the packaging or in the documentation distributed with the product, except for a warning that the “contents are flammable”, which shall always be included on the product label.

NOTE The authority having jurisdiction might require additional labelling such as the appropriate UN identification number and description as defined in the UN Model Regulations on the Transport of Dangerous Goods, part or model number and other cautions and hazard warnings pertinent to the metal hydride MH assembly.

7.2.2 Hazards associated with the solid materials

The manufacturer shall include on the label warnings consistent with the potential hazards of the materials contained within the MH assembly. Consideration should include hazards from reactivity with air, water or other fluids.

7.2.3 Labelling concerning removable valve protection

When required by 6.2.7.4, labelling shall include the following, “WARNING: Valve may be damaged if subjected to impact. KEEP VALVE PROTECTION IN PLACE WHEN NOT CONNECTED FOR USE.”

7.2.4 Temperature Warning Labelling

The manufacturer shall include on the label a warning: “DO NOT EXPOSE TO TEMPERATURES ABOVE xx °C, OPEN FLAMES OR IGNITION SOURCES.”, where xx shall be no greater than the maximum service temperature.

8 Documentation accompanying the product

8.1 Material safety data sheets

The material safety data sheets (MSDS) covering both the hydrogen gas and the contained hydrogen absorbing alloy shall be provided for inclusion with all product shipments. The MSDS shall include safety and handling requirements to be followed in case of hydrogen leakage and/or breach of the storage system, exposing the hydrogen absorbing alloy and any potential reactivity with substances such as air, water and cooling fluids, if applicable.

8.2 User's or operating manual

8.2.1 General

A user's or operating manual shall be provided by the manufacturer. The user's or operating manual shall include the minimum service conditions specified in Clause 4, hydrogen quality, initial fill and refill procedures, disposal and recycling information and/or other pertinent limitations on use, including the minimum ventilation for the in-use and storage locations, the minimum periodic testing and inspection procedures, if applicable.

8.2.2 Initial fill and refill procedures

8.2.2.1 Inspection prior to initial filling and refilling

The manufacturer shall specify inspection procedures to be carried out prior to initial filling and prior to refilling of the MH assembly.

Items to be inspected shall include whether the MH assembly is within its service life, labels are legible and secure, components are not damaged or missing in the interface, and that the shell and valve are not damaged, and have not been tampered with or abused.

Criteria shall be provided as to when refilling is allowed or when an MH assembly shall be removed from service.

8.2.2.2 Charging specifications

The manufacturer shall provide the following information, for the initial filling and refilling of the MH assembly:

— safety precautions and potential hazards of which to be aware;

— method for determining when the rated capacity described in 4.2 has been achieved;

— minimum and maximum pressure range (maximum pressure shall not exceed RCP);

— minimum and maximum temperature range;

— other special conditions required for the initial filling and refilling.

8.2.2.3 Equipment

The manufacturer shall specify the requirements for the equipment to be used for initial filling and refilling of MH assemblies to prevent overcharging.

8.2.2.4 Inspections and checks after initial filling and refilling

The manufacturer shall specify an inspection procedure to be carried out after the initial filling and after refilling of the MH assembly. Items to be inspected shall include leakage of hydrogen from the MH assembly and damaged or missing components in the interface (e.g. damaged threads, O-rings or seals).

8.2.2.5 Periodic inspection and testing

The manufacturer shall specify the minimum periodic inspection and testing requirements. These requirements shall be in accordance with the applicable ISO periodic inspection and test standard for the shell (e.g. ISO 6406, ISO 10461 and ISO 11623). In all cases, the periodicity for the periodic inspection and testing shall not exceed 5 years.

Annex A
(informative)

Material compatibility for hydrogen service

A.1 Material compatibility for hydrogen service

The components in which gaseous hydrogen or hydrogen-containing fluids are processed, as well as all parts used to seal or interconnect the same, should be sufficiently resistant to the chemical and physical action of hydrogen at the operating conditions.

A.2 Metals and metallic materials

The users of this International Standard should be aware that engineering materials exposed to hydrogen in their service environment may exhibit an increased susceptibility to hydrogen assisted corrosion via different mechanisms such as hydrogen embrittlement and hydrogen attack.

Hydrogen embrittlement is defined as a process resulting in a decrease of the toughness or ductility of a metal due to the permeation of atomic hydrogen.

Hydrogen embrittlement has been recognized classically as being of two types. The first, known as internal hydrogen embrittlement, occurs when the hydrogen enters the metal matrix through material processing techniques and supersaturates the metal with hydrogen. The second type, environmental hydrogen embrittlement, results from hydrogen being absorbed by solid metals coming from the service environment.

The atomic hydrogen dissolved within a metal interacts with the intrinsic defects of the metal typically increasing crack-propagation susceptibility, and thus degrading such basic properties as ductility and fracture toughness. There are both important material and environmental variables that contribute to hydrogen-assisted fractures in metals. The material microstructure is an important consideration as second phases, which may or may not be present due to compositional and processing variations, may affect the resistance of the metal to fracture. Second phases, such as ferrite stringers in austenitic stainless steels, may also have a specific orientation leading to profound anisotropic response in the materials. In general, metals can also be processed to have a wide range of strengths, and the resistance to hydrogen-assisted fracture is known to decrease as the strength of the alloy is increased.

The environmental variables affecting hydrogen-assisted fracture include the pressure of hydrogen, temperature, chemical environment and strain rate. In general, the susceptibility to hydrogen-assisted fracture increases as hydrogen pressure increases. The effect of temperature, however, is not as systematic. Some metals such as austenitic stainless steels exhibit a local maximum in hydrogen-assisted fracture susceptibility as a function of temperature. Although not well understood, trace gases mixed with hydrogen gas can also affect hydrogen-assisted fractures. Moisture, for example, may be detrimental to aluminium alloys since wet oxidation produces high-fugacity hydrogen, while in some steels moisture is believed to improve the resistance to hydrogen-assisted fracture by producing surface films that serve as kinetic barriers to hydrogen uptake. A so-called inverse strain rate effect is generally observed in the presence of hydrogen; in other words, metals are less susceptible to hydrogen-assisted fracture at high strain rates.

At temperatures close to ambient, this phenomenon can affect metals with body centred cubic crystal lattice structure, for example ferritic steels. In the absence of residual stress or external loading, environmental hydrogen embrittlement is manifested in various forms, such as blistering, internal cracking, hydride formation and reduced ductility. With a tensile stress or stress-intensity factor exceeding a specific threshold, the atomic hydrogen interacts with the metal to induce sub-critical crack growth leading to fracture.

Hydrogen embrittlement can occur during elevated-temperature thermal treatments, and in service during electroplating, contact with maintenance chemicals, corrosion reactions, cathodic protection, and operating in high-pressure or high temperature hydrogen.

Many low-alloyed structural steels may suffer from hydrogen attack at temperatures as low as 200 °C. This is a non-reversible degradation of the steel microstructure caused by a chemical reaction between diffusing hydrogen and the carbide particles in the steel that results in the nucleation, growth and merging of methane bubbles along grain boundaries to form fissures.

Hydride embrittlement occurs in metals such as titanium and zirconium and is the process of forming thermodynamically stable and relatively brittle hydride phases within the structure.

Clad welding and welds between dissimilar materials often involve high alloy materials. During operation at temperatures over 250 °C, hydrogen diffuses in the fusion line between the high-alloy weld and the unalloyed/low alloy base material. During shutdown, the material temperature drops. The reduced solubility and diffusibility of hydrogen breaks the weld by disbonding.

The following are some general recommendations for managing the risk of hydrogen embrittlement.

— Select raw materials with a low susceptibility to hydrogen embrittlement by controlling the chemistry (e.g. use of carbide stabilizers), microstructure (e.g. use of austenitic stainless steels), and mechanical properties (e.g. restriction of hardness, preferably below 225 HV, and minimization of residual stresses through heat treatment). Use test methods specified in ISO 11114-4 to select metallic materials resistant to hydrogen embrittlement. The API Publication 941 shows the limitations of various types of steel as a function of hydrogen pressure and temperature. The susceptibility to hydrogen embrittlement of some commonly used metals is summarized in ISO/TR 15916.

— Clad welds and welds between dissimilar materials used in hydrogen service should be ultrasonically tested at regular intervals and after uncontrolled shutdowns in which the equipment may have cooled rapidly.

— Minimize the level of applied stress and exposure to fatigue situations.

— When plating parts, manage the anode/cathode surface area and efficiency, resulting in proper control of applied current densities. High current densities increase hydrogen charging.

— Clean the metals using non-cathodic alkaline solutions, and using inhibited acid solutions.

— Use abrasive cleaners for materials having a hardness of 40 HRC or above.

— Use process control checks, when necessary, to mitigate risk of hydrogen embrittlement during manufacturing.

A.3 Polymers, elastomers and other non-metallic materials

Most polymers can be considered suitable for gaseous hydrogen service. Due account should be given to the fact that hydrogen diffuses through these materials much more easily than through metals. Polytetrafluoroethylene [PTFE or Teflon®[1)]] and polychlorotrifluoroethylene [PCTFE or Kel-F®[2)]] are generally

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1) Teflon® is the trade name of a product supplied by DuPont. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) Kel-F® is a registered trade name of 3M Company. In 1996, 3M discontinued manufacturing of Kel-F and, today, all PCTFE resin is manufactured by Daikin under the trade name of Neoflon® or by Allied Signal under the trade name of Aclon®. Kel-F is still the most commonly used trade name used to describe PCTFE. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

suitable for hydrogen service. Suitability of other materials should be verified. Guidance can be found in ISO 11114-2, ISO/TR 15916 and ANSI/AIAA G-095.

A.4 Other references

Further guidance on hydrogen assisted corrosion and control techniques may be found through the following organizations and their standards:

A.4.1 International Organization for Standardization (www.iso.org)

See bibliography [1] to [14].

A.4.2 American Institute of Aeronautics and Astronautics (www.aiaa.org)

See bibliography [15].

A.4.3 American Petroleum Institute (www.api.org)

See bibliography [16] and [17].

A.4.4 American Society for Testing and Materials (www.astm.org)

See bibliography [18] to [32].

A.4.5 American Society of Mechanical Engineers (www.asme.org)

See bibliography [33] to [35].

A.4.6 American Welding Society (www.aws.org)

See bibliography [36].

A.4.7 ASM International (www.asminternational.org) and Society of Automotive Engineers (www.sae.org)

See bibliography [37] to [39].

A.4.8 National Association of Corrosion Engineers (www.nace.org)

See bibliography [40] and [41].

Annex B
(normative)

Environmental tests

B.1 Exposure to fluids

B.1.1 General

This test is applicable to MH assembly shells comprised of Type II and III fibre-wrapped cylinders.

Two shells shall be tested in a condition representative of installed geometry including coating (if applicable), brackets and gaskets, and pressure fittings using the same sealing configuration (i.e. O-rings) as that used in service.

The two shells are subjected to preconditioning in accordance with B.1.2 and then exposed to a sequence of environments, pressures and temperatures in accordance with Table B.1. Although preconditioning and fluid exposure is performed on the cylindrical section of the shell, all of the shell, including the domed sections, shall be as resistant to the exposure environments as are the exposed areas. As an alternative, a single cylinder approach may be used in which both the immersion test and the other fluid exposure test may be carried out on one cylinder as indicated in Table B.1. In this case, care shall be taken to prevent cross contamination among the fluids.

B.1.2 Preconditioning

B.1.2.1 Preconditioning apparatus

The following types of apparatus shall be used for preconditioning the test shell by pendulum and gravel impact.

The pendulum impact apparatus shall comprise:

a) a steel impact body having the shape of a pyramid with equilateral triangle faces and a square base, the summit and the edges being rounded to a radius of 3 mm;

b) a pendulum, the centre of percussion of which coincides with the centre of gravity of the pyramid; its distance from the axis of rotation of the pendulum being 1 m and the total mass of the pendulum referred to its centre of percussion being 15 kg;

c) a means of determining that the energy of the pendulum at the moment of impact is not less than 30 N·m and is as close to that value as possible;

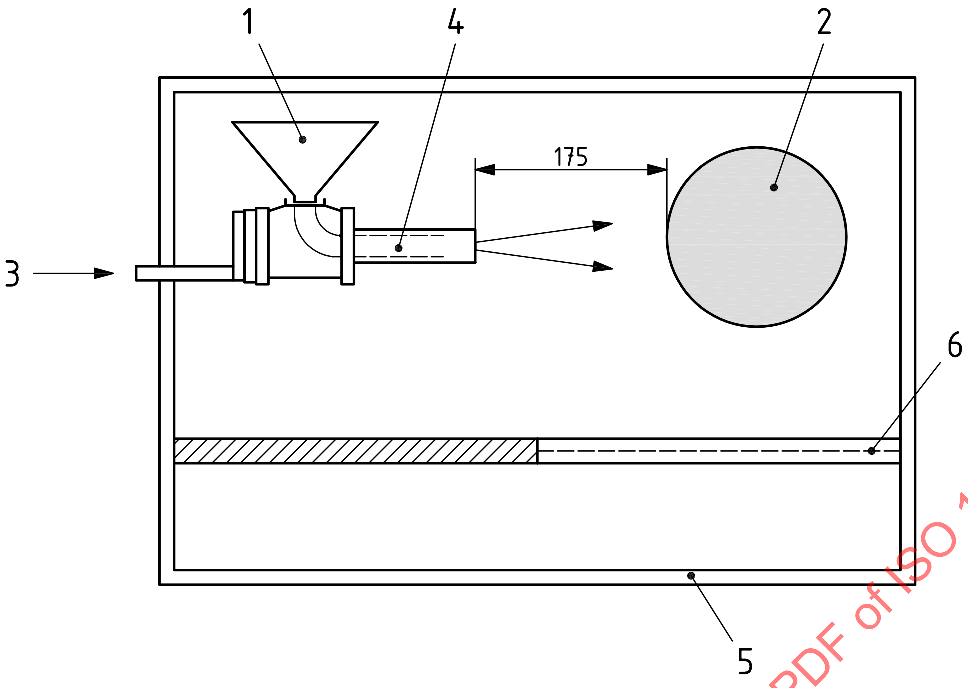
d) a means of holding the shell in position by the end bosses during impact.

The gravel impact machine shall comprise:

a) an impact machine, constructed according to the design specifications shown in Figure B.1 and capable of being operated in accordance with ASTM D3170 except that the shell may be at ambient temperature during gravel impact;

b) gravel, comprising alluvial road gravel passing through a 16 mm space screen but retained on a 9,5 mm space screen. Each application shall consist of 550 ml of graded gravel (approximately 250 to 300 stones).

Dimensions in millimetres



Key

1 funnel
2 shell under test
3 air inlet
4 50 mm pipe
5 cabinet approximately 500 mm wide
6 sizing screen

Figure B.1 — Gravel impact machine

B.1.2.2 Preconditioning procedure

B.1.2.2.1 Preconditioning for the immersion test

Preconditioning by both pendulum impact and gravel impact shall be carried out on the portion of the shell to be used for the immersion test (see B.1.3.1).

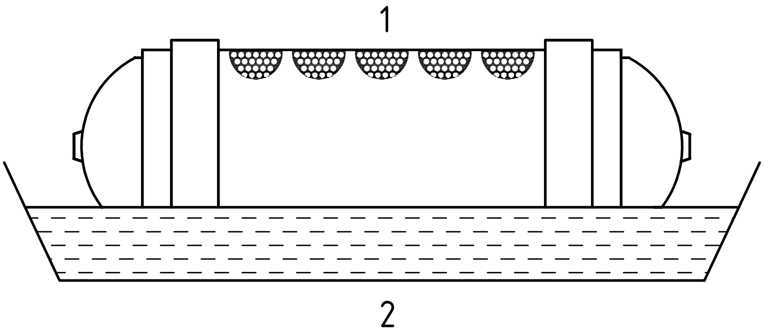
With the shell unpressurized, precondition the central section of the shell that will be submerged, by an impact of the pendulum body at three locations spaced approximately 150 mm apart. Following the pendulum impact, precondition each of the three locations by gravel impact application. Additionally, precondition by a single impact of the pendulum body a location within the submerged portion of each domed section and 50 mm (measured axially) from the tangent.

B.1.2.2.2 Preconditioning for the other fluid exposure test

Preconditioning by gravel impact only shall be carried out on the portion of the shell to be used for the other fluid exposure test (see B.1.3.2).

Divide the upper section of the cylinder used for the other fluid exposure test into five distinct areas of a nominal diameter 100 mm and mark these for preconditioning and fluid exposure (see Figure B.2). Ensure that the areas do not overlap on the shell surface. If the single shell approach is used, also ensure that these areas do not overlap with the section of the shell that will be subjected to the immersion test. While convenient for testing, the areas need not be oriented along a single line.

With the shell unpressurized, precondition each of the five marked areas identified as per the above instructions (see Figure B.2) for the other fluid exposure test by gravel impact application.



Key

1 other fluid exposure area

2 immersion area (lower third)

Figure B.2 — Cylinder orientation and layout of exposure areas

B.1.3 Test conditions

B.1.3.1 Immersion test

At the appropriate stages in the test sequence (see Table B.1), orient the shell horizontally to immerse the lower third of the shell diameter in a simulated acid rain/road salt water solution composed of the following compounds:

— deionized water;

— a mass fraction of (2,5 ± 0,1) % of sodium chloride;

— a mass fraction of (2,5 ± 0,1) % of calcium chloride;

— sulfuric acid in sufficient quantity to achieve a solution pH of 4,0 ± 0,2.

Adjust the solution level and pH prior to each step of the immersion test.

Maintain the temperature of the bath at (21 ± 5) °C. During immersion, hold the unsubmerged section of the shell in ambient air.

B.1.3.2 Other fluid exposure

At the appropriate stages in the test sequence (see Table B.1), expose each marked area to one of five test solutions described below . Use the same test solution for each location throughout the test.

— an aqueous solution with a minimum volume fraction of 19 % of sulfuric acid;

— an aqueous solution with a minimum mass fraction of 25 % sodium hydroxide;

— a volume fraction of 30 % methanol in gasoline;

— an aqueous solution with a minimum mass fraction of 28 % ammonium nitrate;

— an aqueous solution with a minimum volume fraction of 50 % methyl alcohol (i.e. windscreen washer fluid).

