ISO TC 197 N 358

Date: 2006-09-08

ISO/PDTS 16111

ISO TC 197/WG 10

Secretariat: SCC

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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Document type: Technical Specification
Document subtype:
Document stage: (30) Committee
Document language: E

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Foreword

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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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

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ISO/TS 16111 was prepared by Technical Committee ISO/TC 197, *Hydrogen technologies.*

Introduction

As the utilization of gaseous hydrogen evolves from the chemical industry into a fuel for various emerging 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 later released and used under specific thermodynamic conditions. This technical specification will describe the service conditions, design criteria, type tests, and routine tests for these canisters.

Transportable gas storage devices — Hydrogen absorbed in reversible metal hydride

1 Scope

This technical specification defines the requirements applicable to the safe design and use of transportable hydrogen gas storage canisters including all necessary shut-off valve, pressure-relief devices (PRD), and appurtenances, intended for use with reversible metal hydride, hydrogen storage systems. This technical specification only applies to refillable storage canisters where hydrogen is the only transferred media. Storage canisters intended to be used as fixed fuel storage onboard hydrogen fuelled vehicles are excluded.

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

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

ISO 14687, *Hydrogen fuel — Product specification*

ISO 16528, *Boilers and pressure vessels*

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
canister
single complete hydrogen storage system, including shell, metal hydride, PRD, shut-off valve and other appurtenances (e.g. for heat exchange, to prevent excessive stress on the shell walls due to hydride expansion, etc)

NOTE The canister extends only to, and includes the first shut-off valve.

3.3
design stress limit
total stress loading allowed on the shell wall, according to the standard to which the shell was designed

3.4
full flow capacity pressure
gas pressure at which the pressure relief device is fully open

3.5
hydrogen absorbing alloy
material capable of combining directly with hydrogen gas to form a reversible metal hydride

3.6
maximum developed pressure
MDP
highest gas gauge pressure for a canister at rated capacity and equilibrated at the maximum service temperature

NOTE The MDP term was specifically selected for metal hydride systems to avoid confusion with the MAWP and the service pressure used in other ISO standards. In metal hydride systems, the shell design needs to take into account the gas pressure plus the pressure exerted by the hydrogen absorbing alloy expansion.

3.7
metal hydride
solid material formed by reaction between hydrogen and hydrogen absorbing alloy

3.8
normal operating conditions
range of conditions such as pressure, temperature, hydrogen flow rate, hydrogen impurities, etc that the product may be exposed to during all use and filling operations

3.9
normal service conditions
range of conditions, such as pressure, temperature, hydrogen flow rate, hydrogen impurities etc. that the product may be exposed to during normal operating, transportation and storage conditions

3.10
pressure relief device
PRD
basic safety device used to relieve excessive pressure within the canister before damage to the canister can occur

NOTE A pressure relief device may be "pressure-activated", set to activate at a certain pressure. Alternately, 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".

3.11
pressure relief valve
PRV
PRD that includes a valve that will open at a set pressure and reclose once the pressure drops below a set pressure

NOTE Pressure relief valves are typically spring-loaded valves.

3.12
rated capacity
stated deliverable quantity of hydrogen specified by the manufacturer

3.13
rated charging pressure
RCP
maximum pressure allowed to be applied to the product for refilling

3.14
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.

3.15
rupture
structural failure of a shell resulting in the rapid and violent release of the stored energy in such a manner that it may pose a safety hazard to people or property

3.16
shell
enclosure designed to contain the hydrogen gas, metal hydride and other internal components of the canister

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

3.17
stress level at MDP
sum of all the stresses on the shell wall caused by the metal hydride material at rated capacity, hydrogen gas at MDP and any other applicable mechanical loadings

3.18
transportable
designed to be mobile and not intended to be used in a fixed, permanent installation

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.

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 canister, and other applicable mechanical loadings.

4.1.4 PRD activation pressure

The pressure of actuation of pressure-activated PRD shall be specified by the manufacturer and shall be greater than the MDP. For pressure-relief valves (PRV), the full flow capacity pressure shall also be specified.

4.2 Rated capacity

The manufacturer shall state the rated capacity of the canister 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 canister 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 canister 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.

4.3.3 PRD activation temperature

The temperature at which any thermally actuated PRD is set to activate shall be specified by the manufacturer and it shall be greater than the maximum service temperature. The PRD shall have a pressure rating of greater than the MDP at all temperatures less than or equal to 10°C above the maximum service temperature.

NOTE Exposure to higher temperatures may be expected in some geographical regions and should be considered.

4.4 Environmental conditions

The canisters 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 canister was designed.

4.5 Service life

The service life for the canisters 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 5.1 and in no case shall exceed 20 years.

4.6 Requalification procedures

The canister may be requalified in accordance with the requirements of the standard to which the shell was originally designed, or in accordance with a method acceptable to the authority having jurisdiction.

NOTE Caution should be taken, as certain procedures (e.g. hydrostatic testing) allowed for cylinder or pressure vessel requalification may not be appropriate for metal hydride canisters. In such cases, an alternative method (e.g. ultrasonic examination) may be applicable.

If requalification is authorized, the manufacturer shall specify the minimum requalification procedures.

4.7 Hydrogen quality

The quality of the hydrogen gas that shall be used to fill a canister 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, the manufacturer may consider including the information on the product label.

4.8 Special service conditions

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

5 Design considerations

5.1 Shell design

The canister shell shall be designed according to ISO 7866, ISO 9809-1, ISO 9809-3, ISO 11119-1, ISO 11119-2 or standards registered in accordance with ISO 16528, as applicable, or as required by the authority having jurisdiction. The shell shall not exceed 150 litres water capacity, and the MDP shall not exceed 25 MPa. The stress level at MDP, including contributors listed in 5.2, shall be less than or equal to the design stress limit allowed by the standard to which the shell is designed (e.g. the shell's maximum allowable working pressure or maximum permissible working pressure). The operating and service temperature ranges for the canister shall be less than or equal to that of the standard to which the shell is designed.

Alternatively for canisters with an internal volume of less than 0,12 litres, the shell design shall be deemed to be appropriate if the canister design meets all the other requirements of this Technical specification, successfully passes all tests specified in Clause 6 and meets the following design criteria:

a) The pressure in the canister shall not exceed 5 MPa at 55 °C when the canister is filled to its rated capacity; and

b) The canister design shall withstand, without leaking or bursting, a minimum shell burst pressure of 2 times the pressure in the canister at 55 °C when filled to rated capacity, or 200 kPa more than the pressure in the canister at 55 °C when filled to rated capacity, whichever is greater.

5.2 Design strength

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

- the MDP;
- thermal stress, including dissimilar rates of thermal expansion and contraction;
- weight of internals in any possible canister 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 canister design shall be subjected to the hydrogen cycling and strain measurement test described in 6.5.

NOTE The process of introducing and subsequently removing hydrogen in the hydrogen absorbing alloy 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 canister 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 canister. The magnitude of the expansion/contraction phenomena will vary greatly as a function of the hydrogen absorbing alloy used.

5.3 Material selection

5.3.1 General

The canister components shall be made of materials that are suitable for the range of conditions expected during normal service conditions over the service life of the canister. 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.

Metal hydride material that is capable of rapid disassociation or explosion when exposed to prolonged heating shall not be used in a canister.

5.3.2 External surfaces

The canister 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 test methods acceptable to the authority having jurisdiction. Any coatings applied to canisters 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 canister.

5.3.3 Compatibility

The compatibility of canister materials with process fluids and solids, specifically embrittlement due to the exposure to hydrogen, shall be considered. Materials necessary for the pressure containment and structural integrity of the canister and its internal and external appurtenances shall be resistant to hydrogen embrittlement, hydrogen attack and reactivity with contained materials and maintain their required integrity for the service life of the canister. 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. Alternatively, materials known to be resistant to hydrogen embrittlement may be used.

If charged with gases or materials that are capable of combining chemically with each other or with the canister material, the materials shall be selected so as the combination does not endanger the canister 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.

5.3.4 Temperature

The canister materials shall be suitable for the normal service temperature range of 4.3.2.

5.4 Overpressure and fire protection

The canister shall be protected with one or more PRD of the self-destructive type, such as fusible triggers, rupture disks and diaphragms, or of the re-sealable type, such as spring-loaded PRV. The canister and any added component (e.g. insulation or protective material) shall collectively pass the fire test specified in 6.2. PRD shall be approved to a standard acceptable to the authority having jurisdiction.

For canisters with an internal volume of less than 0,12 litres and that meet the alternative shell requirements of 5.1, another means may be used to protect from overpressurization, such as venting through a feature integral to the shell. Canisters that use an alternative means of relieving pressure shall meet the acceptance criteria of the fire test specified in 6.2.

5.5 Shut-off valves

5.5.1 General

The canister assembly shall incorporate a shut-off valve that shall close when the assembly is disconnected from the refill or gas-consuming equipment. The shut-off valve shall be required to conform to an applicable standard.

For canisters with an internal volume of less than 0,12 litres and that meet the alternative shell requirements of 5.1, a valve design that does not conform to a standard may be used, if there is not an applicable valve standard. Such canisters shall meet the acceptance criteria of all tests in Clause 6.

All canisters shall provide a means of valve protection.

NOTE Due to the temperature/pressure characteristics of metal hydrides, the development of sub-ambient pressures are possible within canisters, therefore valve selection should include verification that valve seal is maintained with vacuum conditions within the canister.

5.5.2 Integral valve protection

A canister design that uses an integral method of valve protection that is not meant to be removed for canister operation, such as the use of a shroud, collar or recessing the valve in the canister assembly, shall meet the requirements of the drop test in 6.3.

5.5.3 Removable valve protection

The canister designs that use a removable means of valve protection that is meant to be removed for canister operation, such as a cover or cap, shall meet the requirements of the drop test in 6.3 with the protective means in place and meet the requirements of the shut-off valve impact test in 6.6 without the protective means in place.

5.6 Actively cooled canisters

Canisters 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 canister and the cooling system. The cooling system shall be employed when performing the hydrogen cycling and strain measurement test in 6.5.

5.7 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 canisters shall meet the requirements of the hydrogen cycling and strain measurement test of 6.5.

6 Type/qualification tests

6.1 General

The following type tests shall be performed to qualify a canister design. The canister used for the type tests shall be representative of production canisters. The data for all type tests shall be acquired using calibrated instruments.

Procedures shall be put in place to ensure the consistent loading of the hydrogen absorbing alloy in the canister. Any change in shell design, hydrogen absorbing alloy, manufacturing process or installation procedure shall require repeating the fire test of 6.2, the drop test of 6.3 and the hydrogen cycling and strain measurement test of 6.5.

6.2 Fire test

6.2.1 General

The fire test shall be performed on all new canister designs to demonstrate that the fire protection system, such as PRD and/or integral thermal insulation, will prevent the rupture of the canister under the specified fire conditions. Any significant change to the design (for example changes in diameter or length, PRD, shell-type, 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 are not considered significant (i.e. reduction in shell diameter, reduction in shell length, or increase in PRD flow capacity), to show that a new design does not require repeating the fire test.

Precautions shall be taken to ensure safety of personnel and property during the fire test in the event that a canister rupture occurs.

6.2.2 Sample preparation

The canister shall be filled to rated capacity with hydrogen.

6.2.3 Data monitoring and recording

The temperature and pressure of the canister shall be monitored remotely and recorded at intervals of 15 seconds or less. A manual valve shall be installed to allow venting of the canister 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:

— canister manufacturer;

— canister part or model number;

— unique identifier;

— PRD-type and rating;

— PRD location and orientation;

— date of test;

— canister RCP;

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

— canister 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 canister designs that preclude monitoring 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. Additional safety precautions may be required to safely carry out the fire test.

6.2.4 Test set-up, fire source and test method

The fire tests shall be conducted on at least three canisters in each orientation of intended use and/or transportation. For canister designs for which the orientation of use and transportation are not specified, at least three canisters shall be fire tested in each of the vertical and horizontal orientation and any other orientation due to asymmetry of the canister 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 degrees away from the fire source.

The canisters, over their entire width, shall be subjected to a fire source of a maximum length of 1,65 m. For canisters less than 1,65 m in length, the fire source shall totally engulf the canister. Canisters 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 a canister is longer than 1,65 m and is fitted with a PRD at one end, the opposite end of the canister shall be subjected to the fire source. If the canister is fitted with PRD at both ends, or at more than one location along the length of the canister, the fire source shall be centred midway between the PRD that are separated by the greatest horizontal distance.

For canisters less than 0,30 m in length, a temperature-indicating device shall be installed within 0,05 m of, but not in contact with, the canister surface near each end. For canisters longer than 0,30 m, 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).

Canisters shall be subjected to a direct flame impingement test. Sufficient fuel shall be supplied to ensure a burn time of at least 20 minutes. The canister shall be placed in the test orientation with the canister 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 canister. Shielding shall be used to prevent direct flame impingement on tank 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 minutes. 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 canister is reproducible.

NOTE Canisters that have been subjected to the cycling and strain measurement test of 6.5 may be used in this test.

6.2.5 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 failure of the shell, a 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 canister 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 of all canisters subjected to the fire test vent each canister to zero internal gauge pressure without rupture of the canister; or,
- all canisters subjected to the fire test withstand the fire for a minimum of 20 minutes without rupture.

6.3 Drop test

6.3.1 General requirements

All canister designs shall meet the requirements of the drop test. Any significant change to the design, for example changes in shell-type or means of solid particulate containment, shall necessitate repeating the drop test.

The surface onto which the canisters are dropped shall be a smooth horizontal concrete pad or flooring. The container shall be allowed to bounce on the concrete pad or flooring after the initial impact. No attempt shall be made to prevent this secondary impact. A guide rail for posture maintenance may be used.

6.3.2 Sample preparation

The canisters used for these tests shall include their shut-off valve protection in accordance with 5.5. The canisters shall have an equivalent weight (± 2 %), packing density and internal structure as production canisters. Ballast material may be used in place of the hydrogen absorbing alloy. The canisters shall not be pressurized.

6.3.3 Test procedure

Canisters shall be drop tested in accordance with the following conditions. One canister may be used for all drop tests.

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

b) One canister 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 canister is at a height of less than 0,6 m, the drop angle shall be changed to maintain the lower end of the canister 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 canister, the canister shall be dropped at a 45° angle on its weakest end.

c) One canister shall be dropped horizontally from a height of 1,8 m onto a steel apex as shown in Figure 1. The canister 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 canister, the steel apex shall be fixed to the concrete pad or flooring. The canister shall strike the steel apex before striking the concrete pad or flooring.

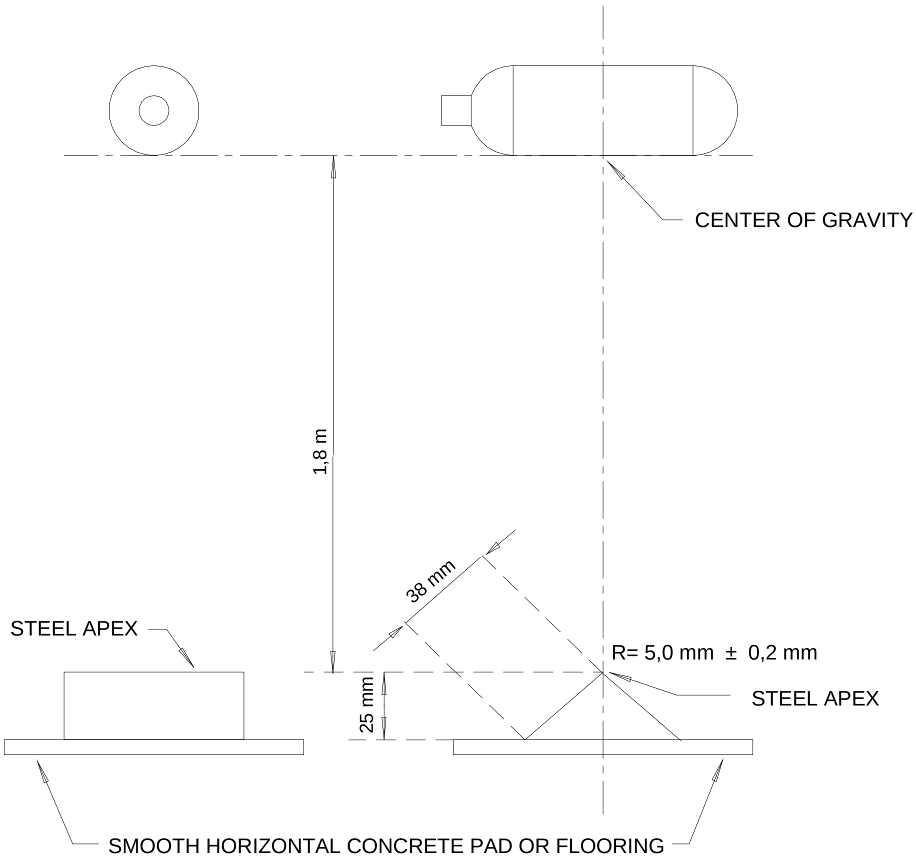

Figure 1 — Canister drop test onto an apex

6.3.4 Acceptance criteria

The shut-off valve shall be intact and function properly after all drop tests.

All canisters that have undergone the drop tests shall be visually inspected and all apparent damage recorded. All canisters shall be subjected to the leak test of 6.4 and then be hydrostatically pressurized to destruction. The canisters shall meet the acceptance criteria of 6.4 and the recorded burst pressures shall exceed 85 % of the minimum shell burst pressure specified by the standard to which the shell was designed or in the case of canisters with an internal volume of less than 0,12 litres, 85 % of the minimum shell burst pressure specified in 5.1

6.4 Leak test

The canister 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
15 ℃ ± 5 ℃	RCP
Maximum service temperature	MDP

An acceptable result shall be a total hydrogen leak rate of less than 10 standard cm^3/h (standard conditions of 0 ℃ and 101,325 kPa absolute). If hydrogen gas is not used, the leak rate shall be converted into an equivalent hydrogen leak rate.

NOTE Before placing the canister in an enclosed area to perform the leak test, 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.

6.5 Hydrogen cycling and strain measurement test

6.5.1 General

The hydrogen cycling and strain measurement test shall be performed on all new canister designs to demonstrate that the design stress limits of the shell are not exceeded during use. Any significant change to the design (including, but not limited to changes to: the shell type, shell specifications, means of solid particulate containment or hydrogen absorbing alloy) shall necessitate repeating the hydrogen cycling and strain measurement test. Canisters 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 shall be taken to ensure safety of personnel and property during testing in the event that a canister failure or hydrogen release occurs.

6.5.2 Test set-up

Each canister shall be adequately instrumented with strain gauges to determine the maximum local strain that the shell experiences during cycling. With metal hydride canisters, the strain may not be uniform throughout the canister. The number and location of the strain gauges required to ensure that the highest strain experienced by the shell may be determined from engineering models based on knowledge of the design, including the internal configuration and geometry, hydrogen absorbing alloy distribution, etc. If engineering models cannot accurately determine the points of expected highest strain, then the number and locations of required strain gauges shall be empirically determined by extensively instrumenting at least two canisters 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 canisters, bending strain shall be monitored on flat sections of canisters 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 canister 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 type II, III and IV fibre-wrapped composite tanks) 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 canisters with hydrogen.

6.5.3 Test method

For canisters designed to be transported and used in a single orientation, at least five canisters shall be tested in that orientation. For canister designs that do not preclude use in more than one orientation, at least three canisters shall be tested in two orientations perpendicular to each other, with the canister axis horizontal and vertical. The canisters 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 50 cycles and until the acceptable results defined in 6.5.4 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 50 cycle intervals, with the canisters charged to not more than 5 % of their rated capacity, each canister 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 minutes. This cycle shall be repeated 12 times for a total of 3 hours for each canister. 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 canisters with a mass greater than 100 kg, the following vibration sequence may be used as an alternate 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 min ± 5 min.

6.5.4 Acceptance criteria

For each strain gauge in a period of at least 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 canister 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:

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

where:

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

x is the cycle number,

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

N the number of consecutive cycles analysed and not to be less than 50,

y the measured strain, and

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

The canister shall be cycled until, for a period including not less than 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. This criterion shall be met by all strain gauges on a canister for the same period of consecutive cycles.

Additionally, after completion of the cycling and strain measurement test, all canisters shall be leak tested at 15 °C ± 5 °C, pressurized to RCP and meet the accep tance criteria of the leak test of 6.4. At least one canister from each orientation tested shall be subjected to the fire test of 6.1 and meet the acceptance criteria.

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

6.6 Shut-off valve impact test

6.6.1 General

As indicated in 5.5, canister designs that employ a removable means of valve protection shall be subjected to the following valve impact test.

6.6.2 Sample preparation

Three canisters shall be subjected to this 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 canisters shall not be pressurized with gas during the test. The removable shut-off valve protection shall be removed for this test.

6.6.3 Test procedure

A hardened steel ball with a Brinell hardness of 248 ± 3 shall be used as the impact object or impact ball. Each canister and the impact ball shall be conditioned for at least 4 hours at –40 °C, and within 5 minutes after conditioning shall be subjected to two impacts as follows. Each canister shall be rigidly anchored and the impact ball shall be allowed to free fall from a height calculated to impart to the side of the shut-off valve the energy specified in Table 2. After the first impact, the shut-off valve and canister assembly shall be rotated 180° and a second side impact test shall be conducted.

Table 2 — Ball impact requirements for valves

Canister type (*V* = internal volume in litres)	**Minimum energy (E)**[a] Joules
V ≤ 0,35 litres	1,02
0,35 litres < *V* ≤ 10 litres	6,80
10 litres < *V* ≤ 25 litres	13,50
25 litres < *V* ≤ 100 litres	27,10
100 litres < *V*	162,70

[a] $E = mg_c h$

where:

E is energy, expressed in joules (J);

m is mass of the impact ball, expressed in kilograms (kg);

g_c is the Earth's gravitational constant (9.8 m/s^2);

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

6.6.4 Acceptance criteria

Following the two impacts tests, each shut-off valve and canister assembly shall be visually inspected for damage and subjected to the leak test of 6.4 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.

If leakage or damage is observed following the impact test, the test shall be repeated on three canisters fitted with their removable shut-off valve protection. If the three canisters meet the acceptance criteria, the design shall be considered as acceptable provided each canister is marked in accordance with 8.2.3.

6.7 Type test reports

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

7 Routine tests and inspections

The manufacturer shall perform routine tests and inspection on each canister and maintain records for not less than 10 years or 1,5 times the service life of the canister, whichever is longer. The routine tests and inspections shall include (at least) the following:

— Each completed canister shall be subjected to a leak test at 15 °C ± 5 °C, pressurized to RCP and meet the acceptance criteria of the leak test of 6.4.

— For all shells used in the manufacturing of canisters the documentation verifying that the shell was manufactured, tested and qualified in accordance to the shell standard shall be obtained and maintained by the canister manufacturer. The canister manufacturer shall perform incoming inspection of shells to the degree necessary to ensure that the shell meet the specified requirements.

8 Marking, labelling, and documentation

8.1 Marking

The canister shall be stamped or permanently marked with:

a) a reference to this international standard;

b) the RCP in megapascals (MPa);

c) the manufacturer's identification;

d) the date of manufacturing (year in four digits and month in two digits);

e) a manufacturer’s serial or unique identification number;

f) the date of expiry based on the maximum service life (year in four digits and month in two digits); and,

g) other information required by the authority having jurisdiction.

NOTE 1 In cases where the RCP is less than 1 MPa, the RCP shall be marked with up to two decimal places (0,XX).

NOTE 2 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 instead, with the exception of b) RCP, e) unique identifier, and f) date of expiry, which shall be marked.

8.2 Labelling

8.2.1 General

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

NOTE 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.

8.2.2 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 canister. Consideration should include hazards from reactivity with air, water or other fluids.

8.2.3 Labelling concerning removable valve protection

When required by 6.6.4, labelling shall include the following, “WARNING: Valve may be damaged if subjected to impact. KEEP VALVE PROTECTION IN PLACE WHEN NOT CONNECTED FOR USE.”

9 Documentation accompanying the product

9.1 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, cooling fluids, etc, if applicable.

9.2 Users or operating manual

9.2.1 General

A users or operating manual shall be provided by the manufacturer. The users 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 requalification procedures, if applicable.

9.2.2 Initial fill and refill procedures

9.2.2.1 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 canister.

Items to be inspected shall include whether the canister 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 a canister shall be removed from service.

9.2.2.2 Charging specifications

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

— safety precautions and potential hazards to be aware of;

- a 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.

9.2.2.3 Equipment

The manufacturer shall specify the requirements for the equipment to be used for initial fill and refilling of canisters.

9.2.2.4 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 canister. Items to be inspected shall include leakage of hydrogen from the canister and damaged or missing components in the interface (e.g. damaged threads, O-rings or seals).

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 technical specification 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 ℃. 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/DIS 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 in non-cathodic alkaline solutions, and in 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 easier than through metals. Polytetrafluoroethylene (PTFE or Teflon®[1]) and Polychlorotrifluoroethylene (PCTFE or Kel-F®[2]) are generally suitable for hydrogen service.

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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 & 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.

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.ch)

ISO 2626:1973, *Copper — Hydrogen embrittlement test*

ISO 3690:2000, *Welding and allied processes — Determination of hydrogen content in ferritic steel arc weld metal*

ISO 7539-6:2003, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

ISO 9587:1999, *Metallic and other inorganic coatings — Pretreatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588:1999, *Metallic and other inorganic coatings — Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 11114-4:2005, *Transportable gas cylinders — Compatibility of cylinders and valve materials with gas contents — Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement*

ISO 15330:1999, *Fasteners — Preloading test for the detection of hydrogen embrittlement — Parallel bearing surface method*

ISO 15724:2001, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

ISO 17081:2004, *Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique*

ISO/TR 15916:2004, *Basic considerations for the safety of hydrogen systems*

A.4.2 American Institute of Aeronantics and Astronautics (www.aira.org)

ANSI/AIAA G-095-2004, *Guide to Safety of Hydrogen and Hydrogen Systems*

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

API RP 934-2000, *Materials and Fabrication Requirements for 2 ¼ Cr-1 Mo & 3Cr-1Mo Steel Heavy Wall Pressure Vessels for High Temperature, High Pressure Hydrogen Service*

API RP 941-2004, *Steels for Hydrogen Service at Elevated Temperatures and Pressures in Petroleum Refineries and Petrochemical Plants*

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

ASTM B577-93 (2004) $^{\epsilon 1}$, *Standard Test Methods for Detection of Cuprous Oxide (Hydrogen Embrittlement Susceptibility) in Copper*

ASTM B83904, *Standard Test Method for Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners, and Rod-Inclined Wedge Method*

ASTM B849-02, *Standard Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement*

ASTM B850-98 (2004), *Standard Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement*

ASTM E168103, *Standard Test Method for Determining a Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials*

ASTM F326-96 (2001) $^{\epsilon 1}$, *Standard Test Method for Electronic Measurement for Hydrogen Embrittlement from Cadmium-Electroplating Processes*

ASTM F519-05, *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments*

ASTM F1459-93(1998) $^{\epsilon 2}$, *Standard Test Method for Determination of the Susceptibility of Metallic Materials to Gaseous Hydrogen Embrittlement*

ASTM F1624-00, *Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique*

ASTM F1940-01, *Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners*

ASTM F2078-01, *Standard Terminology Relating to Hydrogen Embrittlement Testing*

ASTM G129-00, *Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking*

ASTM G142-98 (2004), *Standard Test Method for Determination of Susceptibility of Metals to Embrittlement in Hydrogen Containing Environments at High Pressure, High Temperature, or Both*

ASTM G146-01, *Standard Practice for Evaluation of Disbonding of Bimetallic Stainless Alloy/Steel Plate for Use in High-Pressure, High-Temperature Refinery Hydrogen Service*

ASTM G148-97 (2003), *Standard Practice for Evaluation of Hydrogen Uptake, Permeation, and Transport in Metals by an Electrochemical Technique*

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

2004 ASME Boiler and Pressure Vessel Code

ASME B31.1-2004, *Power piping*

ASME B31.3-2004, *Process piping*

A.4.6 American Welding Society (http://www.aws.org/)

ANSI/AWS A4.3-93 (R1997), *Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding*

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

SAE/AMS 2451/4-2002, *Plating, Brush, Cadmium Corrosion Protective, Low Hydrogen Embrittlement*

SAE/AMS 2759/9-2003, *Hydrogen Embrittlement Relief (Baking) of Steel Parts*

SAE/USCAR 5-1/2, *Avoidance of Hydrogen Embrittlement of Steel*

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

NACE TM0177-2005, *Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H_2S Environments*

NACE TM02842003, *Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking*