



SLOVENSKI STANDARD
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Kriogene posode - Združljivost plin/material

Cryogenic vessels - Gas/material compatibility

Kryo-Behälter - Verträglichkeit von Gas/Werkstoffen

Réipients cryogéniques - Compatibilité entre gaz et matériaux

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English version

Cryogenic vessels - Gas/material compatibility

Réceptifs cryogéniques - Compatibilité entre gaz et matériaux

Kryo-Behälter - Verträglichkeit von Gas/Werkstoffen

This European Standard was approved by CEN on 9 June 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 268, "Cryogenic vessels", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2002, and conflicting national standards shall be withdrawn at the latest by January 2002.

This document replaces EN 1797-1:1998.

For relationship with EU Directives, see informative annex ZA, which is an integral part of this document.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directives.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European Standard specifies requirements for gas/materials compatibility for cryogenic vessels (such as chemical resistance) but it does not cover mechanical properties (e.g. for low temperature application).

It gives guidance for compatibility with gases other than oxygen and it gives detailed requirements for oxygen and oxygen enriched atmosphere compatibility and defines the testing methods for establishing oxygen compatibility of materials (metallic and non-metallic) to be used for cryogenic vessels and associated equipment.

It mainly deals with materials that are normally or could be in contact with liquid/gaseous oxygen e.g., materials for cryogenic vessels used for the storage and/or transport of liquid oxygen.

It also deals with the materials which can be in contact with oxygen enriched environment e.g. insulating materials used for nitrogen, neon, hydrogen and helium cryogenic vessels in case of air condensation.

2 Normative references

This European Standard incorporates by dated or undated references provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publication apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 849: 1996, *Transportable gas cylinder – Cylinder valves - Specification and type testing.*

EN 12300, *Cryogenic vessels - Cleanliness for cryogenic service.*

EN ISO 11114-1, *Transportable gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 1 : Metallic materials (ISO 11114-1:1997).*

prEN ISO 11114-2:1997, *Transportable gas cylinders - Compatibility of cylinder and valve materials with gas contents - Part 2 : Non-metallic materials (ISO/DIS 11114-2).*

3 Compatibility of materials with gases other than oxygen

The cryogenic vessels are used in a range of temperature from very low temperature to ambient temperature. The problems of compatibility with gases other than oxygen such as corrosion, hydrogen embrittlement normally occur at ambient temperature and become negligible at cryogenic temperature.

So, in case of gases other than oxygen, EN ISO 11114-1 and prEN ISO 11114-2:1997 may be used as a guide for cryogenic vessels.

4 General requirements for oxygen service

4.1 Evaluation of materials for oxygen service

The selection of a material for use with oxygen and/or in an oxygen enriched atmosphere is primarily a matter of understanding the circumstances that cause oxygen to react with the material. Most materials in contact with oxygen will not ignite without a source of ignition energy. When an energy input rate, as converted to heat, is greater than the rate of heat dissipation, and the resulting heat increase is continued for sufficient time, ignition and combustion will occur. Thus, two things shall be considered

- the material's minimum ignition temperature ;
- the energy sources that will produce a sufficient increase in the temperature of the material.

These should be viewed in the context of the entire system design so that the specific factors listed below will assume the proper relative significance.

The specific factors are :

- the properties of the materials, including the factors affecting ease of ignition and the conditions affecting potential resulting damage (heat of reaction) ;
- the operating conditions : pressure, temperature, gas velocity, oxygen concentrations and oxygen state (gaseous or liquid), surface contamination in accordance with EN 12300 ;
- the potential sources of ignition (friction, heat of compression, heat from mass impact, heat from particle impact, static electricity, electrical arc, resonance, internal flexing etc.) ;
- the reaction effect (consequence on the surroundings etc.) ;
- additional factors (performance requirements, prior experience, availability and cost).

CAUTION This European Standard specifies the minimum acceptance requirements for materials in oxygen and enriched air service. In the cases of severe conditions and when the operating pressure is above 40 bar, additional tests to those specified should be considered.

The use of materials in cryogenic vessels which do not pass the tests outlined in 4.4.1 and/or 4.4.2 shall be supported by a favourable risk assessment and/or documented evidence of previous long term satisfactory service in use.

4.2 Evaluation of metallic materials

Metallic materials normally used for the construction of cryogenic vessels i.e. low alloy steels, nickel steels, stainless steels, copper and copper alloys, aluminium and aluminium alloys do not normally present any incompatibility when in contact with oxygen.

The cases in which some ignitions or violent reactions may occur are when very thin materials are used with high surface/volume ratio, and when high ignition energy is available e.g. pump failure. Thin materials e.g. thinner than 0,1 mm shall be tested in accordance with 4.4.2 in conditions as close as possible to the real operational conditions (e.g. for multi-layer insulations use similar number of layers and configuration). Materials to be used in applications where the ignition energy is potentially high should be subjected to special consideration.

For cryogenic vessels intended for oxygen service the test shall be performed with oxygen. For cryogenic vessels intended for nitrogen, hydrogen or helium service, when materials are located in an area where contact with condensed enriched air is a risk, the test described in 4.4.2 shall be performed with cryogenic O₂/N₂ mixtures containing at least 50 % oxygen.

4.3 Evaluation of non metallic materials

Non metallic materials include, for example, plastics, elastomers, lubricants, ceramics, glasses and glues. Some of these materials present a high risk of ignition when in contact with oxygen and should be avoided or carefully selected and used in limited quantity.

Some fully oxidised materials such as ceramics and glass present no risk of ignition provided they are not contaminated.

Any non metallic materials, other than fully oxidised materials, in contact with liquid oxygen shall be tested in accordance with 4.4.1 and 4.4.2. Consideration shall be given to testing materials used in those parts of the system where liquid oxygen accumulation may incidentally occur (e.g. in the insulation).

For cryogenic vessels intended for oxygen service the test shall be performed with oxygen. For cryogenic vessels intended for nitrogen, hydrogen or helium service, when materials are located in an area where contact with condensed enriched air is a risk, the test described in 4.4.2 shall be performed with cryogenic O₂/N₂ mixtures containing at least 50 % oxygen.

Any non metallic materials, other than fully oxidised materials, in contact with gaseous oxygen shall be tested in accordance with 4.4.1. Consideration shall be given to testing materials used in those parts of the system where gaseous oxygen accumulation may incidentally occur (e.g. in the insulation).

4.4 Test methods and acceptance criteria

Each material to be tested shall be clearly identified, normally by the commercial name and the manufacturer's name.

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4.4.1 Ignition tests

Two alternative test methods are described in 4.4.1.1 or 4.4.1.2. The advantages and disadvantages of each are given in annex C.

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Materials not satisfying the requirements of 4.4.1.1 or 4.4.1.2 can still be used providing they successfully pass, in their actual operating configuration, the "Oxygen pressure surge test" described in 5.3.8 of EN 849:1996 (e.g. for a valve sealing material, the entire valve or a representative assembly shall be tested).

4.4.1.1 Spontaneous ignition test ("Bomb test")

4.4.1.1.1 Test procedure

The test procedure is given in annex A.

4.4.1.1.2 Acceptance criteria

The spontaneous ignition temperature determined in accordance with 4.4.1.1.1 shall be not less than the values given in Table 1.

Table 1 — Minimum spontaneous ignition temperature

Maximum working pressure Bar (gauge)	Minimum Spontaneous Ignition Temperature (SIT) °C	Remark
3	200	
10	230	
20	250	
40	300	
100	350	
150	375	Complementary test may be advisable (see 4.1)
207	400	
Above 207 up to 345	400	
NOTE Intermediate values can be determined by linear interpolation.		

4.4.1.2 Pressure surge test

4.4.1.2.1 Test procedure iTeh STANDARD PREVIEW

The test procedure is given in annex B. (standards.iteh.ai)

4.4.1.2.2 Acceptance criteria

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No reaction shall be observed during 5 consecutive pressure surge impacts at the intended maximum working pressure.

4.4.2 Mechanical impact test in liquid oxygen (LOX)

4.4.2.1 Test procedure

The mechanical impact test shall be performed at atmospheric pressure in liquid oxygen generally as described in the bibliography. This is an example of preferred test equipment but the details are not mandatory. The test shall be conducted :

- on material with the surface condition that is intended for use ;
- on material in a physical form delivered for use (i.e. solid, powder etc.) ;
- at an impact energy per unit contact area of 79 J/cm².

4.4.2.2 Acceptance criteria

No reaction shall be detected within a series of 20 tests.

Annex A (normative)

Spontaneous ignition test (Bomb test)

A.1 General

This annex defines a test method to determine the spontaneous ignition temperature of non-metallic materials in pressurised gaseous oxygen.

Spontaneous ignition temperature is a criterion for the comparison and the classification of materials, and can be used as an aid in the choice of materials used in the presence of pressurised gaseous oxygen.

A.2 Principle

A small quantity of the test material is slowly heated in oxygen under pressure. The continuous recording of pressure and temperature is used to determine spontaneous ignition, which is seen as a sudden increase in temperature and pressure.

A.3 Preparation of test pieces

Test pieces shall be prepared by procedures that prevent contamination.

Test pieces can be in liquid or solid form. In the case of solids, the materials shall be cut into a minimum of 6 pieces. The total mass of the pieces used in each test shall be at least 60 mg.

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A.4 Test equipment

Figure A.1 shows the basic principle of the test equipment. When others methods of heating are used the heating rate of the specimen should be less than 20°C/min. If inductively heated furnace are used, the temperature rate can be up to 110°C/min.

A thermocouple inside a glove finger positioned as close as possible to the test piece is used to monitor on a recorder temperature variation with an accuracy of $\pm 2^\circ \text{C}$.

The internal pressure shall be monitored and recorded with an accuracy of ± 2 bar.

The equipment, and in particular, the autoclave, shall be designed to resist violent internal reactions (explosions).

A.5 Oxygen purity

The gas used shall contain not less than 99,5% oxygen. The hydrocarbon content shall be less than 10 ppm by volume.

A.6 Test procedure

The test piece contained in the sample holder is put into the bomb. The bomb is then sealed and purged to remove any air and any possible residual combustion products from previous tests. Oxygen is then introduced at a minimum pressure which will produce at least 120 bar at ignition.

Whilst continuously recording the temperature and pressure, the temperature is raised, at a rate of up to 20°C/min by adjustment of the heating power, to the spontaneous ignition temperature or to a maximum temperature of 500°C.

The spontaneous ignition temperature is indicated on the recording by the sudden increase in both parameters caused by the internal reaction.

A.7 Results

The record of the test is used as shown in Figure A.2 to determine the three parameters, T_i , ΔT and ΔP , where

T_i is the spontaneous ignition temperature ;

ΔT is the increase in temperature at moment of ignition ;

ΔP increase in pressure at moment of ignition.

Materials are classified in accordance with their spontaneous ignition temperature.

Temperature and pressure increase, ΔT and ΔP characterise the violence of the reaction.

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