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**Cryogenic vessels — Cryogenic  
insulation performance**

*Réipients cryogéniques — Performances d'isolation cryogénique*

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## Foreword

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

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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.

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 21014 was prepared by Technical Committee ISO/TC 220, *Cryogenic vessels*.

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## Introduction

Traditionally, there have been different methods of defining the insulation performance of cryogenic vessels. It is therefore necessary to harmonize such methods for different cryogenic vessels.

Figure 1 shows a logic diagram to help in the understanding of this International Standard.

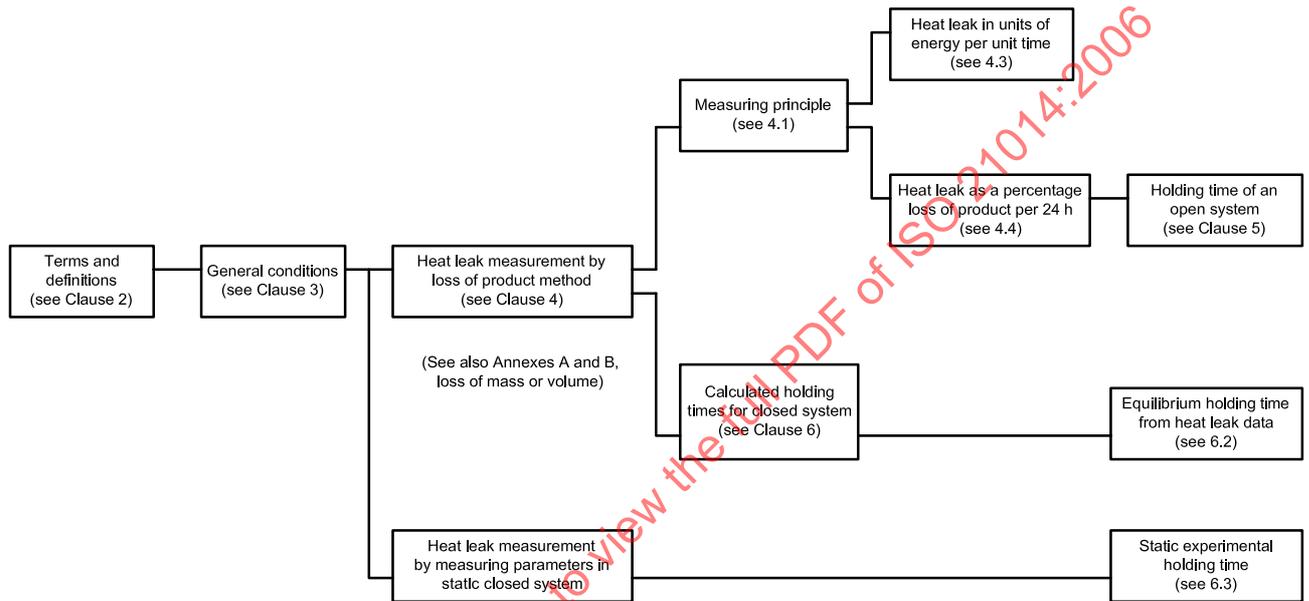


Figure 1 — Logic diagram



# Cryogenic vessels — Cryogenic insulation performance

## 1 Scope

This International Standard defines practical methods for determining the heat-leak performance of cryogenic vessels. The methods include measurement on both open and closed systems.

This International Standard neither specifies the requirement levels for insulation performance nor when the defined methods should be applied. These requirements may be defined in design or operational standards/regulations.

## 2 Terms and definitions

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

### 2.1

#### **open system**

(during test) system kept at a constant pressure (e.g. atmospheric pressure) in which the gas produced by the evaporation of the test fluid is continuously released to atmosphere

### 2.2

#### **closed system**

(during test) system in which the mass of the contents is kept constant with no input or output of product

### 2.3

#### **heat-leak rate**

quantity of heat transferred per unit time from the ambient air to the contents of the inner vessel

NOTE In an open system, the heat leak causes a loss of product; in a closed system, it causes a rise in pressure.

### 2.4

#### **holding time**

(open system) time expected to elapse, for a specified degree of filling, from initial filling level until the vessel is empty (no more liquid) calculated from heat-leak data

### 2.5

#### **holding time**

(closed system) time elapsed, for a specified degree of filling, from establishing the initial filling condition until the pressure has risen, due to heat leak, to the set pressure of the pressure-limiting device

NOTE 1 For transportable vessels, this holding time is determined without the effects of stratification.

NOTE 2 A pressure-limiting device can be a safety valve, a rupture disc, a back-pressure regulator, or any other device installed to limit the system pressure under normal operating conditions.

#### 2.5.1

##### **equilibrium holding time**

holding time calculated from a specified heat leak assuming that liquid and vapour are constantly in equilibrium (without stratification)

### 2.5.2

#### **longest equilibrium holding time**

equilibrium holding time calculated from heat-leak data for a vessel when filled with the quantity of product giving the longest holding time

### 2.5.3

#### **static experimental holding time**

time it takes starting from atmospheric pressure, or from a stated pressure in the case of fluids where the starting pressure cannot be atmospheric pressure (e.g. 10 bar for CO<sub>2</sub>), to reach the set pressure of the pressure-limiting device with the tank initially filled to its maximum allowable filling mass

### 2.6

#### **maximum allowable filling mass**

initial mass that results in the tank becoming hydraulically full (98 % for all fluids except helium and 100 % for helium) at the point that the pressure-limiting device operates

NOTE For fluids in a supercritical condition, the maximum allowable filling mass will be a function of the holding time and will be stated.

## 3 General conditions for all methods

The measurements specified in this International Standard shall be carried out under the following conditions.

**3.1** The cryogenic fluid used for testing shall be agreed upon between the involved parties. Liquid nitrogen may normally be used, except in cases where the vessel to be tested is designed for a specific cryogenic fluid.

**3.2** The liquid and gaseous phases shall be in equilibrium at the beginning of a test. When a test is carried out at a higher pressure than atmospheric pressure, it is important that the liquid equilibrium pressure is not lower than this test pressure.

**3.3** The test environment shall be stable and constant during the test. It shall be as close as possible to the following reference conditions:

- ambient temperature, 15 °C;
- atmospheric pressure, 1 013 mbar.

For products other than carbon dioxide and nitrous oxide:

- vessel reference pressure, 1 013 mbar.

For carbon dioxide and nitrous oxide:

- vessel reference pressure, 15 bar (gauge).

**3.4** The vessel and its contents shall have reached a stable temperature before the beginning of the measuring period. Equilibrium conditions are obtained after a period of stabilization, the duration of which depends on the size of the vessel and the type and configuration of the insulation.

**3.5** All accessories of the vessel which can influence the result of the measurement shall be clearly defined and specified in the report.

**3.6** All instrumentation used shall be verified by calibration.

**3.7** It is not necessary to use the method defined in this International Standard to evaluate the insulation performance resulting from small modifications; this may be done by simple extrapolation.

## 4 Measuring the heat leak by the loss of product method

### 4.1 General

There are two methods of measuring the heat leak:

- direct measurement of loss of mass;
- indirect measurement of loss of mass by measuring the gaseous volumetric discharge rate.

The filling level shall be  $50^{+10}_0$  % of the maximum filling level at the start of measurement, unless otherwise stated.

The ambient temperature, ambient barometric pressure and the operating pressure at the top of the vessel shall be recorded throughout the test so as to be used for correction purposes. The temperature sensor(s) shall be placed in the immediate proximity of the tank being tested, but sited such that they are unaffected directly by cold gas discharged from the vents.

The minimum measurement duration shall be 24 h after stable conditions have been reached.

During the test, precautions shall be taken to avoid agitation of the liquid, except for tanks designed for land transport mode.

When measuring the rate of discharge of gas escaping from the vessel by a flow meter, it is essential that the entire gas flow passes through the meter. The gas flow rate shall be determined as a mass flow rate by using either of the following:

- mass flow meter;
- volumetric flow meter (an appropriate method is shown in Annex A).

### 4.2 Test procedure

The test procedure shall be as follows:

- a) precool the vessel;
- b) leave for a first stabilization period;
- c) adjust the filling to the intended starting level (e.g.  $50^{+10}_0$  %);
- d) connect the instrumentation (e.g. gas flow meter);
- e) leave for a second stabilization period;
- f) take a sufficient number of readings to establish an acceptable thermal equilibrium before the start of the measuring period;
- g) determine the mass of the vessel contents at the start of measuring period, if direct measurement of the mass is used;
- h) record readings for a minimum of 24 h;
- i) determine the loss of product in mass units (when gaseous flow is measured) in accordance with Annex A;
- j) reduce to reference conditions in accordance with Annex B.

### 4.3 Determination of the heat leak in units of energy per unit time

The rate of product loss (kg/s) during the measurement period, corrected to the reference conditions in accordance with Annexes A and B, shall be converted to an equivalent heat leak,  $Q$ , as given in 4.4.

To calculate the heat leak with a product other than the test product, compensation using linear extrapolation in accordance with Annex C may be applied, but only if the difference between the boiling temperature of these products at the reference conditions does not exceed 20 K.

### 4.4 Determination of the heat leak as a percentage loss of product per 24 h

Based on the result obtained in accordance with 4.3, the heat leak as a percentage loss of product per 24 h is calculated as follows.

- a) Correct the measured heat leak to the reference condition for the test product by linear extrapolation, as specified in 4.3.
- b) Calculate the equivalent loss of the test product per day in accordance with the following formula:

$$L = \frac{86\,400 (v_g - v_l) Q}{v_g h_{fg} F} \times 100 \%$$

where

- $F$  is the maximum allowable filling mass of the test product (kg);
- $L$  is the equivalent loss of product as a percentage of  $F$  per day;
- $Q$  is the heat leak (W);
- $h_{fg}$  is the latent heat of vaporization (J/kg) at the vessel reference pressure (see 3.3);
- $v_g$  is the specific volume of vapour (m<sup>3</sup>/kg) at the vessel reference pressure (see 3.3);
- $v_l$  is the specific volume of saturated liquid (m<sup>3</sup>/kg) at the vessel reference pressure (see 3.3);
- 86 400 is the number of seconds per day.

All product-related data shall be taken at correct reference conditions for the specified product. Annex C may be used to determine the equivalent loss of product as a percentage of full tank content per day, for a product other than the test product.

## 5 Determination of the holding time for open systems from heat-leak data

The holding time, in days, for open systems is equal to  $\frac{100}{L}$  for the specified product.

## 6 Holding times for closed systems

### 6.1 Determination of the equilibrium holding time from heat-leak data

The system is in thermal equilibrium, i.e. the liquid and gas phases are saturated and at a temperature corresponding to the saturation pressure at all times. The calculation process shall incorporate correctly the temperature and pressure dependence of the thermodynamic properties. The data source used for calculations shall be identified and the actual value shall be shown in the calculation. Thermodynamic data from bibliography items [1], [2] or [3] may be used. The influence of phase change in the system has to be accounted for in a proper manner.

The thermal mass of the vessel shall be neglected in the calculation, which results in shorter holding times.

For a degree of filling less than that used for the longest holding time, the holding time shall be defined as the time elapsed between when the initial filling condition is established and when the pressure-limiting device opens.

Heat-leak data corrected in accordance with Annex C may be used when different products are concerned.

### 6.2 Determination of the optimum equilibrium holding time from heat-leak data

The equilibrium holding time for a specific product shall be calculated from heat-leak data as follows.

- a) Correct the heat leak,  $Q$ , measured in accordance with Clause 4, to the reference conditions for the specified product by linear extrapolation (see Annex C).
- b) Determine the reference quantity of the specified product as follows.
  - 1) When the critical pressure is greater than the pressure of the pressure-limiting device, the reference quantity is the quantity of product which fills, at operating temperature, 98 % of the volume of the tank below the inlet of the pressure-limiting device under conditions of the pressure-limiting device's recipient opening.
  - 2) When the critical pressure is less than the pressure of the pressure-limiting device, the reference quantity of the product depends directly on the holding time required.
- c) The equilibrium holding time,  $H$ , in hours shall be calculated from the first law of thermodynamics for a constant volume system.

$$H = \frac{(m_{eg} u_{eg} + m_{el} u_{el}) - (m_{ig} u_{ig} + m_{il} u_{il})}{3\,600Q}$$

and

$$m_{eg} = \frac{V - (M \times v_{el})}{(v_{eg} - v_{el})}$$

$$m_{el} = \frac{V - (M \times v_{eg})}{(v_{el} - v_{eg})}$$

$$m_{ig} = \frac{V - (M \times v_{il})}{(v_{ig} - v_{il})}$$

$$m_{il} = \frac{V - (M \times v_{ig})}{(v_{il} - v_{ig})}$$

where

- $V$  is the container gross volume (m<sup>3</sup>);
- $M$  is the mass of contents (kg), as defined in 6.2 b);
- $m_{eg}$  is the mass of vapour at end condition (kg);
- $m_{el}$  is the mass of liquid at end condition (kg);
- $m_{ig}$  is the mass of vapour at initial condition (kg);
- $m_{il}$  is the mass of liquid at initial condition (kg);
- $v_{eg}$  is the specific volume of vapour at end condition (m<sup>3</sup>/kg);
- $v_{el}$  is the specific volume of liquid at end condition (m<sup>3</sup>/kg);
- $v_{ig}$  is the specific volume of vapour at initial condition (m<sup>3</sup>/kg);
- $v_{il}$  is the specific volume of liquid at initial condition (m<sup>3</sup>/kg);
- $u_{eg}$  is the specific internal energy of vapour at end condition (J/kg);
- $u_{el}$  is the specific internal energy of liquid at end condition (J/kg);
- $u_{ig}$  is the specific internal energy of vapour at initial condition (J/kg);
- $u_{il}$  is the specific internal energy of liquid at initial condition (J/kg);
- $Q$  is the heat leak (W) determined in 4.3.

The result can be given in hours or days, whichever is more convenient, and should always be accompanied by a specification of the product referred to and the reference quantity of filling, in kilograms or as a percentage of full vessel capacity or the degree of filling.

### 6.3 Static experimental holding time

The measurement shall be made with the product for which the result is required. Substitute products shall not be used.

The vessel shall be filled to a level exceeding the intended starting level with an amount to allow for a proper stabilization period to reference conditions for the product before the start of the measuring period. When the content level has reached the intended starting level, the system is closed and the recording of the pressure increase started.

The normal level of filling at the start of the measurement shall be as given in 6.2.

If the vessel being tested can not be protected from exposure to sun during the measurement, the result shall still be accepted as relevant provided a remark is made in the test report.

The vessel being tested shall be carefully inspected during the measuring period to ensure that there is no visible leakage of product ("bubble tight":  $10^{-2}$  mbar l s<sup>-1</sup>). If a leakage is observed, the measurement shall be regarded as failed unless the leakage can be measured and found to be less than 1 % of the product per unit time for the vessel being tested under open conditions. In this case, provided the leak can be corrected, the measurement may be continued. A note shall be made in the test report.

During the measuring period, the vessel being tested shall not be moved or otherwise disturbed so that the product is not agitated (to avoid eliminating the influence of stratification unless it is a transportable tank). The result may be given in hours or days, whichever is more convenient, and should always be accompanied by a specification of the product referred to and the reference quantity of filling, in kilograms or as a percentage of full vessel capacity or the degree of filling.

## 7 Test report

The test report shall describe all conditions of the tests. In particular, it shall include

- a) reference to this International Standard;
- b) full identification of the vessel tested and its accessories;
- c) identification of the testing body responsible for the test;
- d) date;
- e) test parameters (particularly when they deviate from the conditions given in this International Standard):
  - 1) cryogenic fluid used for the test;
  - 2) exact filling conditions;
  - 3) recording of ambient (abs) pressure and temperature, noting any special conditions (e.g. exposure to sunshine);
  - 4) full identification and calibration of the instruments used for the test results.

## Annex A (normative)

### Conversion of measured volumetric gaseous flow to mass flow

To convert the measured volumetric gaseous flow to mass flow, it is necessary to take into account the difference between the measuring conditions and specified reference conditions (in accordance with 3.3) of three parameters:

- the reference pressure as prescribed by the flow meter manufacturer (if given) or the pressure at the inlet of the flow meter;
- the temperature of gaseous flow at the inlet of the flow meter;
- the gas density at the reference conditions.

The average gaseous mass flow (kg/s) is calculated with either of the following formulae:

$$Q_m = Q_v \frac{P_a}{1013} \times \frac{288}{T} \times \rho$$

or

$$Q_m = Q_v \times \rho_m$$

where

$Q_v$  is the average measured volumetric gaseous flow ( $m^3/s$ ) at the measuring conditions;

$T$  is the average temperature (K) of the measured volumetric gaseous volume during the measuring period;

$P_a$  is the flow meter reference absolute pressure (mbar) or average of the absolute pressure of the gas at the inlet of the flow meter (mbar) during the measuring period;

$\rho$  is the mass density at 288 K and 1 013 mbar abs ( $kg/m^3$ ). Values of  $\rho$  for several gases addressed by this International Standard are given in Table A.1;

$\rho_m$  is the mass density ( $kg/m^3$ ) at measuring conditions  $T$  and  $P_a$ .

**Table A.1 — Mass density,  $\rho$ , at 288 K and 1 013 mbar abs for different gases**

Product gas	$\rho$ kg/m <sup>3</sup>
Nitrogen	1,185
Oxygen	1,354
Argon	1,691
Helium	0,169
Carbon dioxide	1,874
Nitrous oxide	1,877
Neon	0,853
Xenon	5,58
Krypton	3,55
Hydrogen	0,085

## Annex B (normative)

### Correction of measured mass flow rate with regard to deviation from reference conditions

It is necessary to correct the loss of product measured in accordance with Clause 4 to take into account the influence of variations of

- the ambient temperature;
- the vessel reference pressure;
- the absolute ambient pressure.

#### B.1 Influence of the ambient temperature variations

##### B.1.1 General

The thermal insulation performance of a cryogenic vessel is given for reference conditions in accordance with 3.3.

The rate of heat transfer to the cryogenic fluid is proportional to the temperature difference ( $T_a - T_c$ ),

where

$T_a$  is the absolute ambient temperature;

$T_c$  is the absolute temperature of the vessel contents.

Variation of thermal equilibrium due to variations in  $T_a$  has a time constant (because of delay between the temperature variation and the heat-leak variation) which can be measured. It depends on the vessel design.

##### B.1.2 Practical measurements

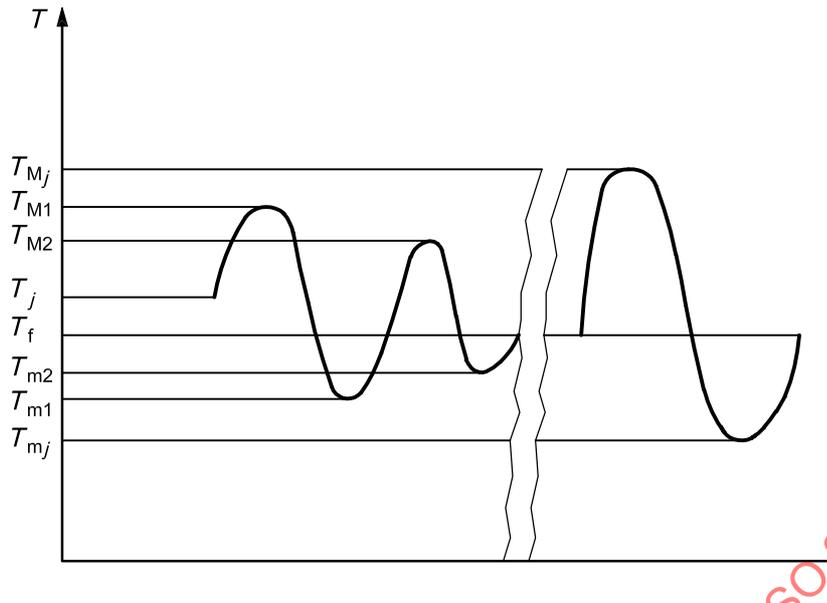
It is necessary to consider two cases.

###### B.1.2.1 Variations of $T_a$ with a 24 h periodicity

To correct the measured loss of product in order to calculate the average loss during 24 h, the average temperature  $T_a$  is used.

Thus, it is sufficient to use 24 h or a multiple of 24 h as the duration of the measurement by loss of volume or as the periodicity of the measurement by loss of mass, in order to avoid making a correction in the time constant.

Temperature variations have the form given in Figure B.1.



**Key**

- $t$  time
- $T$  temperature
- $T_f$  final temperature
- $T_j$  temperature readings
- $T_{Mj}$  ( $j = 1, 2, \dots$ ) maximum temperature readings
- $T_{mj}$  ( $j = 1, 2, \dots$ ) minimum temperature readings

**Figure B.1 — Temperature variations**

A good approximation of the average  $T_a$  during the measuring period is

$$T_a = \frac{T_i + T_f + \sum_{j=1}^{j=n} T_{Mj} + \sum_{j=1}^{j=n} T_{mj}}{2 + 2n}$$

or

$$T_a = \frac{\sum_{j=1}^{j=N} T_j}{N},$$

whichever is higher,

where

$T_j$  is the temperature readings;

$T_i$  is the initial temperature;

$T_f$  is the final temperature;

$T_{Mj}$  is the maximum temperature reading;

$T_{mj}$  is the minimum temperature reading;

$n$  is the number of cycles;

$N$  is the number of readings.

### B.1.2.2 Variations of $T_a$ that are not periodic

This applies, for example, when a prolonged stop in heating of the premises occurs during the measuring period.

To avoid the need to take into account the time constant for thermal equilibrium variations, the loss of product value shall be validated as described in B.1.2.1 only.

### B.1.3 Influence of the pressure variations of the vessel contents

#### B.1.3.1 General

The variation of the pressure,  $P_c$ , of the vessel contents can have the effects described in B.1.3.1.1 to B.1.3.1.3.

**B.1.3.1.1** A supplementary evaporation of liquid if  $P_c$  decreases. This is caused by a lowering of the equilibrium specific enthalpy of the test fluid. The resultant release of energy from the test fluid to achieve equilibrium is available as latent heat of evaporation. In addition, the equilibrium temperature of the liquid decreases, thus increasing the value of  $(T_a - T_c)$  and the resultant heat leak to the liquid.

**B.1.3.1.2** A reduction in evaporation of liquid if  $P_c$  increases. This is caused by an increase in the equilibrium specific enthalpy of the test fluid. The resultant energy absorption from the heat inleak to achieve equilibrium decreases the energy available for evaporation. In addition, the equilibrium temperature of the liquid increases, thus decreasing the value of  $(T_a - T_c)$  and the resultant heat leak to the liquid.

**B.1.3.1.3** The latent heat of evaporation of the vessel contents increases with falling pressure, thus decreasing the rate of evaporation. The opposite condition prevails if the pressure increases.

## B.2 Practical measurements

The average value of vessel contents pressure,  $P_{ca}$ , should be determined as follows:

$$P_{ca} = \frac{P_{ci} + P_{ce}}{2}$$

where

$P_{ci}$  is the equilibrium pressure of contents at start of the test (bar abs);

$P_{ce}$  is the equilibrium pressure of contents at end of the test (bar abs).