INTERNATIONAL STANDARD

ISO 16496

First edition
2016-02-01

Laboratory glassware — Vacuum-jacketed vessels for heat insulation

Verrerie de laboratoire — Récipients à double enveloppe à vide pour isolation thermique

Reference number
ISO 16496:2016(E)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 48, *Laboratory equipment*.

Laboratory glassware — Vacuum-jacketed vessels for heat insulation

1 Scope

This International Standard recommends dimensions and specifies requirements and test methods for laboratory glassware manufactured from borosilicate glass 3.3 and provided with a vacuum jacket for thermal insulation. It covers Dewar vessels, vacuum-jacketed reaction vessels and vacuum-jacketed columns intended for laboratory use and laboratory related applications. Typical dimensions are given in Tables 1 to 5.

This International Standard does not apply to large scale production equipment and equipment operated with pressures of more than 0,1 bar above atmospheric pressure.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 641, *Laboratory glassware — Interchangeable spherical ground joints*

ISO 718, *Laboratory glassware — Thermal shock and thermal shock endurance — Test methods*

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 4803, *Laboratory glassware — Borosilicate glass tubing*

ISO 4790, *Glass-to-glass sealings — Determination of stresses*

3 Terms and definitions

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

3.1
Dewar flask
glass vessel with vacuum jacket for thermal insulation, designed for keeping substances at a controlled temperature within a range from -200 °C to +200 °C

Note 1 to entry: See 8.1 for restrictions on the use of Dewar flasks.

3.2
cryo vessel
vacuum jacketed vessel made of materials other than glass

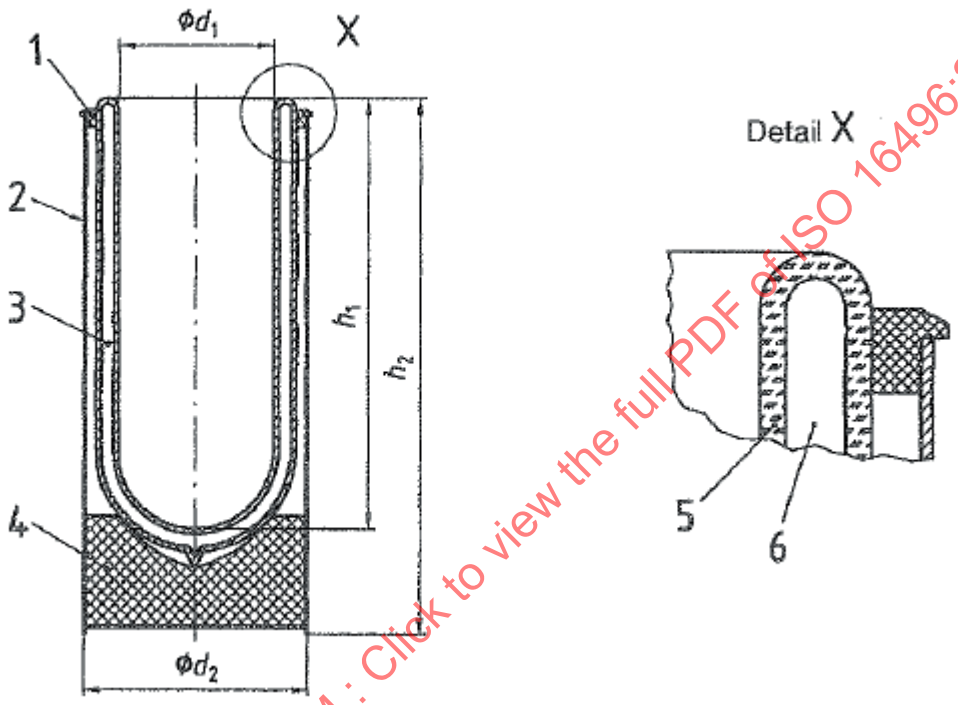
3.3
column
cylindrical vessel for the thermal separation of substances in a laboratory or pilot plant

4 Dimensions

The designs shown in Figures 1 to 5 are for illustrative purposes only. The dimensions given in Tables 1 to 5 are for guidance, other dimensions being permissible, provided the resulting capacities comply with the scope of this International Standard.

4.1 Dewar flasks

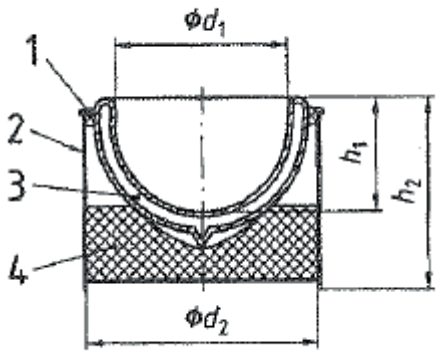
Dewar flasks may have a cylindrical, spherical or dished shape, as shown exemplarily in Figures 1 to 3. Other flask designs are permitted, e.g. flasks with flat bottom or rolled-on or integral flange.



Key

1 soft rubber spacer
2 protective housing
3 Dewar flask
4 plastic foam cushion
5 glass body
6 evacuated space

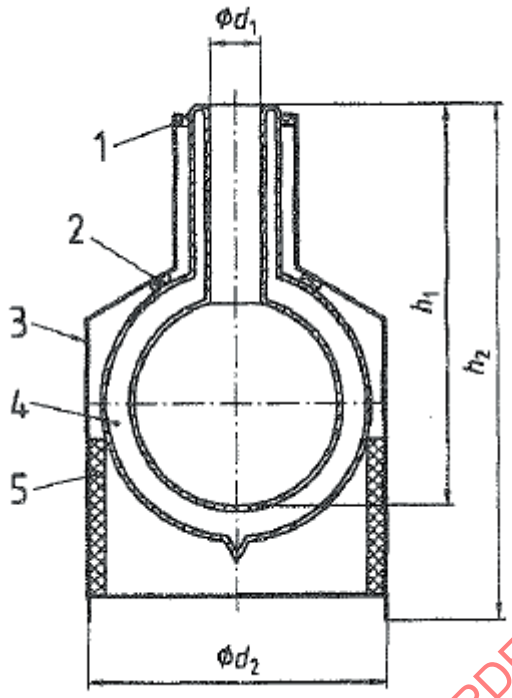
Figure 1 — Cylindrical Dewar flask



Key

1 soft rubber spacer
2 protective housing
3 Dewar flask
4 plastic foam cushion

Figure 2 — Dished Dewar flask



Key

1 soft rubber spacer
2 hard rubber spacer
3 protective housing
4 Dewar flask
5 plastic foam cushion

Figure 3 — Spherical Dewar flask

Table 1 — Dimensions of cylindrical Dewar flasks

Dimensions in millimetres

Nominal capacity	Internal diameter d_1		Internal height h_1		Housing diameter	Overall height
ml	Nominal size	Limit deviations	Nominal size	Limit deviations	d_2[a] ≈	h_2[a] ≈
200	40	±3	170	±3	66	220
500	57		210		82	265
800	67		240		90	300
1 000	77		235		106	300
1 000	100		150		128	190
1 500	90		245		125	305
1 500	100		240		130	300
2 000	90		340		125	395
2 000	100		290		130	350

[a] Dimensions d_2 and h_2 are intended as guideline values.

Table 1 *(continued)*

Nominal capacity ml	Internal diameter d_1		Internal height h_1		Housing diameter d_2[a] ≈	Overall height h_2[a] ≈
	Nominal size	Limit deviations	Nominal size	Limit deviations		
2 000	138	±4	170	±4	168	220
2 500	110		290		140	350
3 000	138		230		170	285
4 000	138		310		170	365
7 000	200	±5	270	±5	240	375
10 000	200		360		240	465
14 000	200		500		240	600
21 000	250	±6	480	±6	295	580
28 000	250		625		295	720
40 000	290		650		340	745

[a] Dimensions d_2 and h_2 are intended as guideline values.

Table 2 — Dimensions of dished Dewar flasks

Dimensions in millimetres

Nominal capacity ml	Internal diameter d_1 ≈	Internal height h_1 ≈	Housing diameter d_2 ≈	Overall height h_2 ≈
260	100	65	130	110
390	110	70	138	115
680	138	80	170	125
1 280	170	110	215	145
4 400	200	140	250	190
8 300	250	170	300	220

NOTE All dimensions are intended as guideline values.

Table 3 — Dimensions of spherical Dewar flasks

Dimensions in millimetres

Nominal capacity ml	Internal neck diameter d_1 ±2	Internal height h_1 ±4	Housing diameter d_2[a] ≈	Overall height h_2[a] ≈
1 000	30	230	175	295
3 000	60	305	225	375
5 000	60	350	260	425
10 000	65	380	330	475

[a] Dimensions d_2 and h_2 are intended as guideline values.

4.2 Reaction vessels

Optional features of reaction vessels include an outlet stopcock at the vessel's bottom, a heating/cooling jacket and a flat flange at the vessel's top.

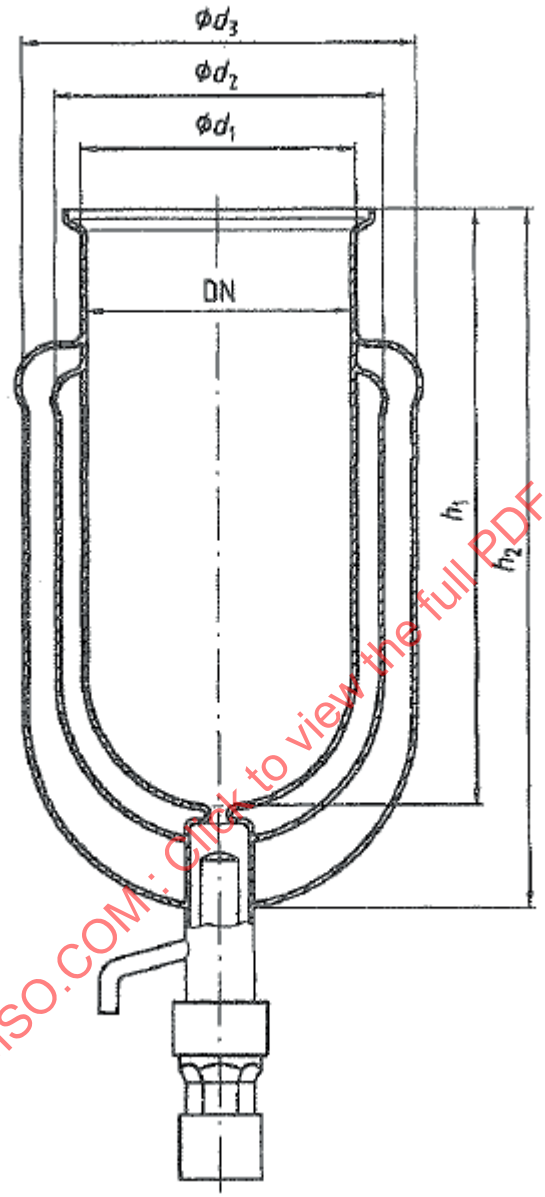


Figure 4 — Example for a reaction vessel with outlet stopcock, heating/cooling jacket and flat flange

4.3 Columns

Columns may be designed to be operated with or without an additional heating jacket. The design of column inlet and outlet connections is at the manufacturer's discretion (see 6.6).

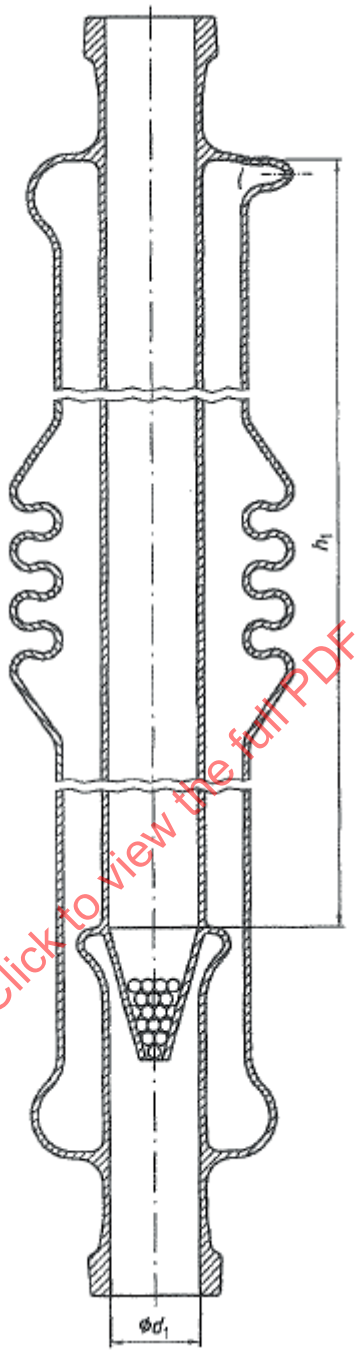


Figure 5 — Column

Table 4 — Dimensions of reaction vessels

Dimensions in millimetres

Nominal capacity ml	Nominal size DN	Internal height h_1 +5 0	Installation height h_2 ±5	Diameters		
				d_1[a]	d_2[a]	d_3[a]
100	60	115	160	70	100	130
250	60	170	215	70	100	130
500	60	260	305	700	100	130
250	100	130	170	105	125	150
500	100	160	200	105	125	150
1 000	100	225	265	105	125	150
1 000	120	205	250	130	160	190
2 000	120	295	340	130	160	190
2 000	150	235	280	160	190	225
3 000	150	290	325	160	190	225
5 000	150	405	450	160	190	225
10 000	150	690	735	160	190	225
5 000	200	290	350	215	250	300
6 000	200	320	380	215	250	300
10 000	200	445	505	215	250	300

[a] Tolerances on d_1, d_2 and d_3 are as specified in ISO 4803 or in national standards. Alternatively, commercial tubing made of borosilicate glass 3.3 may be used.

Table 5 — Dimensions of columns

Dimensions in millimetres

Packing height h_1 ≈	Internal diameter d_1 ≈
300	30 to 85
500	30 to 85
1 000	30 to 85
NOTE The dimensions are intended as guideline values.	

5 Materials

The glass parts of vacuum-jacketed vessels shall be of borosilicate glass 3.3 as specified in ISO 3585, and shall be free of residual strain and defects likely to impair the vessel's appearance, service life and strength.

The protective housing of Dewar flasks shall be made of sheet metal, stainless steel sheet, aluminium or plastics.

6 Construction

6.1 The glass used for the vessel and the glass-to-glass seals shall be free from residual strain likely to impair the strength of the vacuum jacket. Testing shall be performed by examination under polarized light.

6.2 When glass-to-glass seals include borosilicate glass 3.3 of different origins, e.g. where alterations are made to the vessels or in the case of repairs, any residual strain should be determined in accordance with ISO 4790 or as specified in Annex A.

6.3 The vacuum jacket (i.e. all walls that enclose the vacuum) shall have a minimum wall thickness conforming to that calculated in accordance with acknowledged methods given in expert literature. Some examples are listed in the Bibliography. The wall thickness specification does not apply to bellows-type expansion joints of columns. The actual wall thickness shall be determined using an ultrasonic method.

6.4 The outer glass surface of vacuum-jacketed vessels may be coated with plastics for protection against mechanical damage during handling, transport and in the event of implosion.

6.5 Reaction vessel ports and connections should preferably be equipped with flat flanges in accordance with regional or national standards. Other ports and connections are acceptable. The design of connections to the heating or cooling jacket is at the manufacturer's discretion.

6.6 In the case of columns, the design of connections at the column inlets and outlets is at the manufacturer's discretion. If conical or spherical ground joints are used, they shall conform to ISO 383 and ISO 641 or the respective national standards. Flat flanges should comply with regional or national standards.

7 Safety requirements and testing

7.1 General

7.1.1 The walls of glass parts shall have no defects likely to impair their strength, e. g. stones, bubbles, knots, cords, cracks, scratches. Testing shall be performed by visual inspection.

7.1.2 Glass tubing used to fabricate vacuum vessels shall conform to ISO 4803 or to national standards.

7.1.3 Glass-to-glass seals between the inner and outer part of vacuum jackets shall be homogeneous and free from stones, bubbles and sharp edges. Glass-to-glass seals and sealed-off parts shall have a clean and rounded finish. Testing shall be performed by visual inspection.

7.2 Dewar flasks

7.2.1 Dewar flasks supplied for end-use in the laboratory shall have a rigid protective housing made of a material in accordance with Clause 5. Alternatively, the Dewar flask may be coated with plastics, as specified in 6.4.

The glass part and protective housing shall stand upright on a flat horizontal surface without rocking or rotating.

The glass body shall be protected against damage, e. g. by a cushioning between the glass body of the vessel and the protective housing. The cushion (see Figures 1 and 2) shall provide sufficient support so that the tip of the sealed evacuation nozzle does not contact the protective housing when the flask is used as intended.

7.2.2 The glass vessel shall have thermal shock endurance

a) of at least 100 °C in the temperature range from +20 °C to +200 °C or

b) of at least 180 °C in the temperature range from +20 °C to -200 °C.

Test for both temperature ranges, as specified in ISO 718, with the Dewar flasks being filled with the liquid to 30 mm below the top rim, instead of being immersed in the liquid.

7.2.3 Dewar flasks shall not have any gastight closure mechanisms, such as expansion closures or locking closures. Lids shall be designed to ensure equilibration of pressure with the atmosphere.

7.2.4 If applicable, Dewar flasks which are specifically designed to store combustible liquefied gases shall conform to the stipulations of national regulations for these substances.

7.3 Reaction vessels

The sealed evacuation nozzle of reaction vessels shall be protected from mechanical damage, assuming the intended use of the vessel. Plastic protective caps may be useful for this purpose. The fused shoulder shall be rounded without sharp edges. Testing shall be performed by visual inspection.

7.4 Columns

The jacket tubes of columns shall be fitted with internal bellows-type expansion joints. The number of expansion bellows will depend on the length of the column and the intended operating temperature range.

NOTE When columns are used with temperatures >200 °C, it might be useful to employ an additional heating jacket to avoid thermal stresses.

The sealed evacuation nozzle of columns shall be protected from mechanical damage, assuming intended use of the vessel. Plastic protective caps may be useful for this purpose. Testing shall be performed by visual inspection.

8 Use of vacuum vessels

8.1 Safety instructions

When working with vacuum-jacketed vessels, applicable national safety regulations for the work with vacuum shall be observed.

Before use, the vessels shall be visually inspected to ensure that there are no defects, such as chipping, cracks and scratches. Damaged glass parts shall not be used.

A replacement of the glass body of Dewar flasks should be performed by the manufacturer or by qualified personnel. New glass bodies shall not be installed into corroded or deformed protective housings.

Loose lids or stoppers for closure on Dewar flasks shall be designed to ensure pressure equilibration with the atmosphere.

NOTE 1 Dewar flasks are unsuitable for storing or transporting liquefied helium.

NOTE 2 Dewar flasks used to store combustible liquefied gases need to be specifically made for this purpose.

NOTE 3 There are national safety regulations in some countries prohibiting the use of liquefied air or liquefied oxygen for low temperature cooling.

8.2 Functional requirements

Every three to five years, the user should check vacuum jackets of laboratory equipment for the insulation ability. Reaction vessels and columns shall be checked following consultation with the manufacturer.

When testing Dewar flasks as described below, the leakage rate measured by the user shall not exceed the values given by the manufacturer in the user information by more than 15 %. If the values are higher, the user should have the flask re-evaluated by the manufacturer.

In the test, the flask shall be filled to its nominal capacity with liquid nitrogen or dry ice (following the manufacturer's instructions) and placed on a balance. The leakage rate is the weight loss, in grams per day, up to the time at which the vessel contents have evaporated completely.

9 User information

Each vacuum-jacketed vessel shall be accompanied by a user information, which shall comprise of the following:

a) a reference to the appropriate safety instructions in national regulations;

b) operating parameters:

- minimum and maximum operating temperatures;
- permissible pressure in the effective volume;
- with reaction vessels and columns, the minimum permissible heating and cooling times in the specified temperature range;
- with Dewar flasks, leakage rate and restrictions on use, e. g. regarding liquid helium and liquefied combustible gases.

With reaction vessels and columns intended to be assembled to form a more complex apparatus, information on assembly shall be given.

10 Marking

10.1 Dewar flasks

Dewar flasks shall be permanently marked at an appropriate place with the following details:

a) the name and/or trademark of manufacturer and/or supplier;

b) the glass type or brand;

c) the nominal capacity or type;

d) the number of this International Standard, i.e. ISO 16496.

10.2 Reaction vessels and columns

The glass body of vessels and columns shall be marked in as permanent a manner as possible, as follows:

a) the name and/or trademark of manufacturer and/or supplier;

b) the glass type or brand;

c) the type or individual component number;

d) the maximum operating temperature;

e) the number of this International Standard, i.e. ISO 16496.

Those columns which have been individually tested shall also be marked with the test temperature and date of testing.

Reaction vessels shall additionally be marked with their nominal capacity and year of manufacture.