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**Determination of carbon content in  
uranium dioxide powder and sintered  
pellets — High-frequency induction  
furnace combustion —  
Titrimetric/coulometric/infrared absorption  
methods**

ISO 9891:1994

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*Détermination de la teneur en carbone dans la poudre et les pastilles  
frittées de dioxyde d'uranium — Combustion dans un four électrique à  
induction — Méthodes par titrimétrie/coulométrie/absorptiométrie  
infrarouge*



## Foreword

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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.

International Standard ISO 9891 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

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# Determination of carbon content in uranium dioxide powder and sintered pellets — High-frequency induction furnace combustion — Titrimetric/coulometric/infrared absorption methods

## 1 Scope

This International Standard specifies titrimetric/calometric/infrared absorption methods for determining the carbon content in uranium dioxide powder and sintered pellets, the test sample being heated in an induction furnace.

It is applicable to the determination of 5 µg to 500 µg of carbon in uranium dioxide powder and pellets. Interference from sulfur and halogens is prevented by the use of appropriate traps.

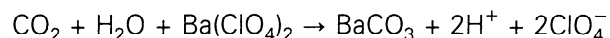
## 2 Principle

A portion of the test sample is heated at a temperature of at least 1 100 °C to 1 200 °C in a quartz-encapsulated tungsten crucible or in a platinum crucible, in an oxygen atmosphere. The evolved oxidation products are passed over a purification trap, filled with manganese dioxide and silver permanganate catalyst. Manganese dioxide absorbs nitrogen oxides. Silver permanganate catalyst will oxidise carbon monoxide to carbon dioxide and absorb sulfur oxides and halogens.

The carbon dioxide is trapped in an absorption solution of barium perchlorate, adjusted to pH = 10,0. Absorption of carbon dioxide causes a decrease of the pH. The initial pH is restored continuously by the addition of hydroxyl ions either by potentiostatic titrimetry or by coulometry.

Alternatively, the carbon dioxide may be determined by absorption of infrared radiation and integration of the signal obtained.

## 3 Reaction



## 4 Reagents and materials

During the analyses, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

**4.1 Oxygen**, of commercial grade, better than 99,9 % (V/V) purity.

**4.2 Carbon dioxide**, commercial grade, better than 99,9 % (V/V) purity.

**4.3 Copper oxide**, 1 mm to 2 mm granules.

**4.4 Soda lime**, of the self indicating type, 1 mm to 2 mm granules.

**4.5 Molecular sieve 4A**, 1,6 mm (1/16 in) pellets, preheated at 300 °C.

**4.6 Silver permanganate catalyst** (in Europe available as Korbl's combustion catalyst), for element analysis.

**4.7 Manganese dioxide**, activated, combustion analysis grade, 0,5 mm to 1,5 mm granules.

**4.8 Accelerators**, tin metal, powder form, low in carbon, or tungsten granules.

**4.9 Moist hydrogen reduced iron**, chips, containing less than 5 ppm of carbon.

**4.10 SRM steels<sup>1)</sup>**, of certified reference material grade, chips, certified for carbon, containing 15 ppm to 500 ppm carbon.

**4.11 Barium perchlorate**  $[\text{Ba}(\text{ClO}_4)_2]$ .

**4.12 2-Propanol** ( $\text{CH}_3\text{CHOHCH}_3$ ).

**4.13 Absorption solution**

Dissolve 200 g of barium perchlorate (4.11) in water. Add 1,0 ml of 2-propanol (4.12), dilute to 1,0 l and mix.

**4.14 Sodium hydroxide, solution** [ $c(\text{NaOH}) = 0,10 \text{ mol/l}$ ], free from carbon dioxide, standardized against potassium hydrogen phthalate.

**4.15 Sodium hydroxide, solution** [ $c(\text{NaOH}) = 0,02 \text{ mol/l}$ ], free from carbon dioxide. Prepare fresh daily by accurate dilution of the sodium hydroxide solution 4.14.

NOTE 1 Use this sodium hydroxide solution (4.15) for the titration of less than 50  $\mu\text{g}$  of carbon, otherwise use the 0,10 mol/l solution (4.14).

## 5 Apparatus

Typical dimensions are given for the apparatus; an alternative system giving similar performances may however be used.

**5.1 High-frequency induction furnace**, 1,5 kVA to 2,5 kVA, 1 MHz to 20 MHz, with silica combustion tube of inner diameter 30 mm (see figure 1). The induction coil surrounding the combustion tube should be 40 mm to 60 mm high and have 4 to 5 turns.

The crucible containing the test portion and, if necessary, an accelerator is supported on an alumina pedestal and positioned within the induction coil to ensure effective coupling.

This furnace gives a temperature of 1 100 °C; an alternative giving a higher temperature may be used.

**5.2 Oxygen purification and flow control system** (see figure 2)

**5.2.1 Oxygen purification system** (see figure 3), consisting of a stainless steel tube, of inner diameter 30 mm and length 200 mm, filled with copper oxide (4.3) and heated at 650 °C. The stainless steel tube is connected to a glass tube, of inner diameter 30 mm and length 300 mm, filled with approximately 100 mm of soda lime (4.4) and approximately 200 mm of molecular sieve (4.5). The layers are separated by layers of quartz wool.

**5.2.2 Pressure regulators 0 kPa to 200 kPa, manometers 0 kPa to 200 kPa, flow regulators and flow meters 0 ml/min to 100 ml/min**

All connections of the gas delivery system should be made using stainless steel tubing of inner diameter 3 mm. The connections between the furnace and the measuring system should preferably be made of stainless steel of inner diameter approximately 1 mm, except for a short piece of PTFE tubing of inner diameter approximately 0,4 mm which is used as the gas inlet to the absorption vessel.

**5.2.3 Two-litre buffer vessel**, to supply additional oxygen during the combustion of steels.

**5.2.4 Six-way valve**, to supply an oxygen backflush to the combustion tube when it is open. This flush prevents absorption of carbon dioxide from the air in the combustion tube.

1) Standard Reference Steels are available from:

NBS (National Bureau of Standards), Washington, D.C., USA;

BCR (Bureau communautaire de référence), Brussels, Belgium;

BAS (Bureau of Analysed Samples), Middlesbrough, United Kingdom;

BAM (Bundesanstalt für Materialprüfung), Berlin, Germany;

IRSID (Institut de recherches de la sidérurgie française), Saint-Germain-en-Laye, France.

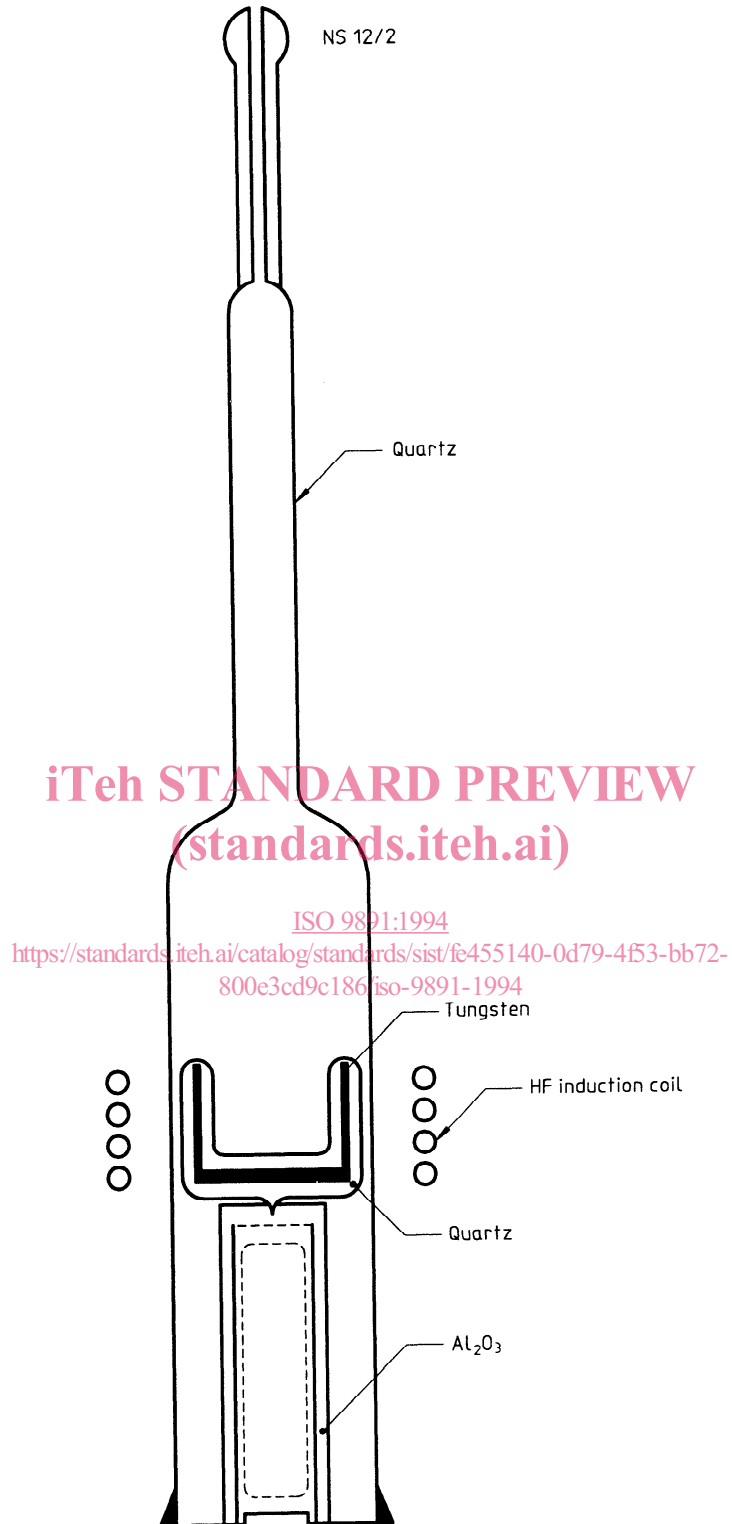
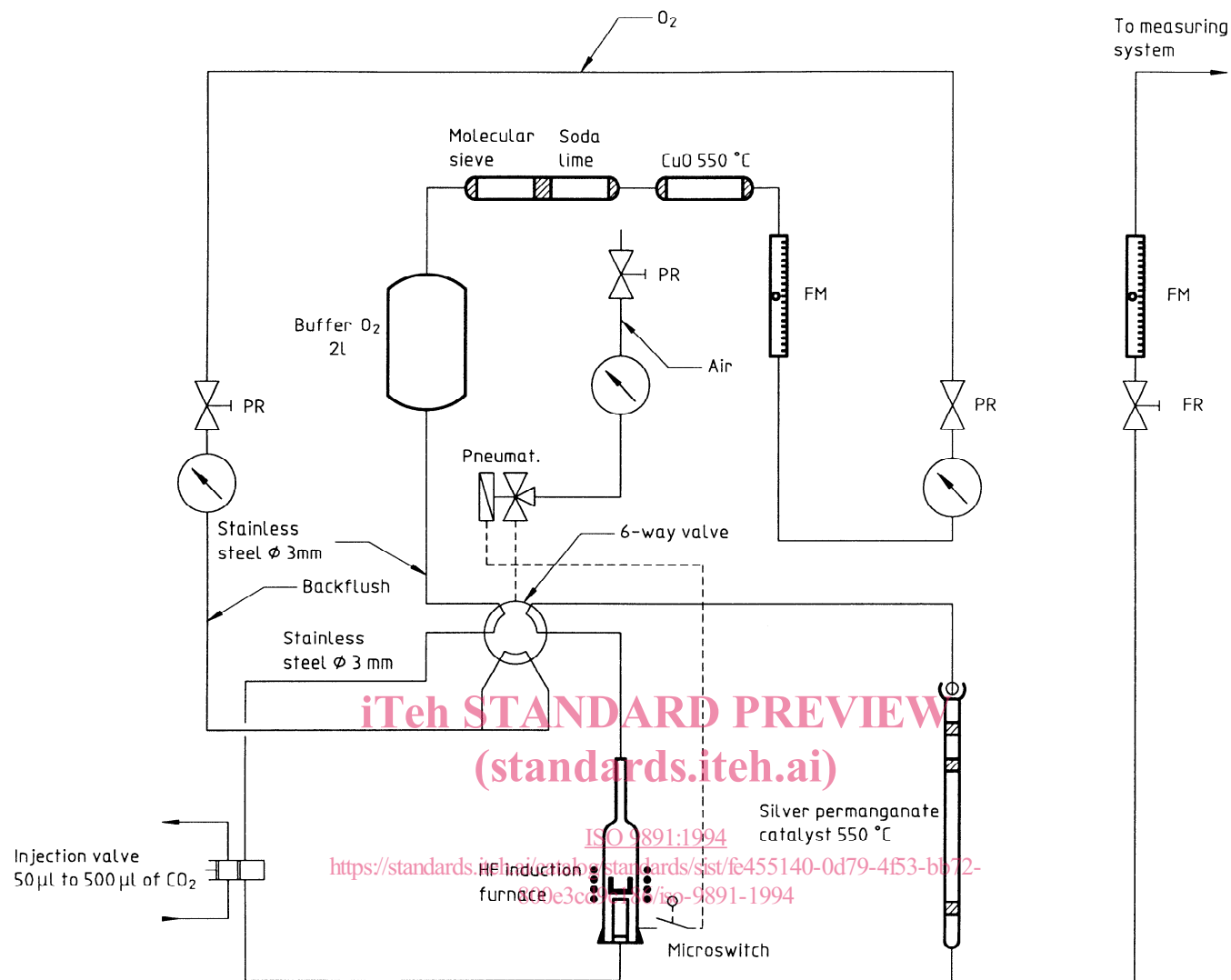


Figure 1 — Combustion tube



- FR = Flow regulator
- PR = Pressure regulator
- FM = Flow meter

Figure 2 — Oxygen purification and flow control system

**5.3 Combustion gas purification tower** (see figure 4), comprising a quartz tube 200 mm long, of inner diameter 8 mm, filled with 40 mm of manganese dioxide (4.7) and 100 mm of silver permanganate catalyst (4.6), the layers separated by layers of quartz wool.

The section containing the silver permanganate catalyst should be heated at 550 °C.

**5.4 Gas dosing valves**, calibrated volumes of 50 µl and 500 µl for the admission of carbon dioxide.

## 5.5 Detection system

**5.5.1 For titrimetry:** titration vessel (see figure 5) or equivalent, temperature controlled at  $25,0\text{ °C} \pm 0,2\text{ °C}$ , filled with absorption solution (4.13), glass (NaCl, AgCl) Ag electrode combination and a potentiostatic control circuit to drive a 1 ml plunger-type burette. Intimate contact between the gas and liquid phase should be provided by efficient stirring with a stirring disc.

**5.5.2 For coulometry:** commercially available cell (see figure 6). Efficient stirring with a stirrer basket and temperature control at  $35,0\text{ °C} \pm 0,2\text{ °C}$  should be provided.

**5.5.3 For infrared absorption:** instrument capable of measuring the amount of specific infrared absorption by carbon dioxide. It consists of an infrared emitter, a sample, a reference cell and a detector. The measured absorption is usually amplified, integrated and converted to give a digital display of the amount of carbon.

**WARNING — Fluoride in more than trace amounts can damage the infrared detector.**

**5.6 Crucibles**, ceramic, platinum or quartz encapsulated tungsten.

## 6 Test sample

**6.1 Uranium dioxide powder:** no preparation is necessary.

**6.2 Uranium dioxide pellets:** crush or break the laboratory sample without producing excessive amounts of fine particles and pass it through a 1 mm sieve until about 5 g has passed.

Retain this portion for use as the test sample.

NOTE 2 Uranium dioxide powder absorbs carbon dioxide; this interferes with the determination. It is removed prior to analyses (see note 6 in 7.4.3).

## 7 Procedure

### 7.1 Setting-up of the apparatus

**7.1.1** Adjust the pressure within the oxygen supply system to about 125 kPa and the oxygen outlet flow to 100 ml/min. After equilibration check both flow meters; the inlet flow and the, adjusted, outlet flow must be equal. If not, trace and repair leaks.

**7.1.2** Place a quartz encapsulated tungsten or a platinum crucible (5.6) on the pedestal, close the combustion tube.

**7.1.3** Fill the absorption vessel with the absorption solution (4.13), stir vigorously and adjust the pH to 10,0. Titrate over 4 min periods until the titrant consumption, in millilitres or coulombs, becomes stable or, in the case of infrared detection, until the integrator output is constant.

NOTE 3 In case of very low carbon contents, it will be advantageous to use pH = 9,5 as the preset potential; at this pH the absorption capacity is less but sensitivity is enhanced.

### 7.2 Functional check

**7.2.1** Heat the crucible at 1 100 °C to 1 200 °C for 4 min periods until the volume of titrant ( $V_0$ ) or number of coulombs ( $C_0$ ) is constant. In case of infrared detection, compensate the output to zero until stable.

#### NOTES

4  $V_0$ ,  $C_0$  or the compensated signal should be equivalent to not more than 5 µg of carbon.

5 Quartz encapsulated tungsten or platinum crucibles can be used only if the powder of the HF generator can be controlled and if a proper temperature measuring device is available. Too much power will of course cause melting of these materials.

Dimensions in millimetres

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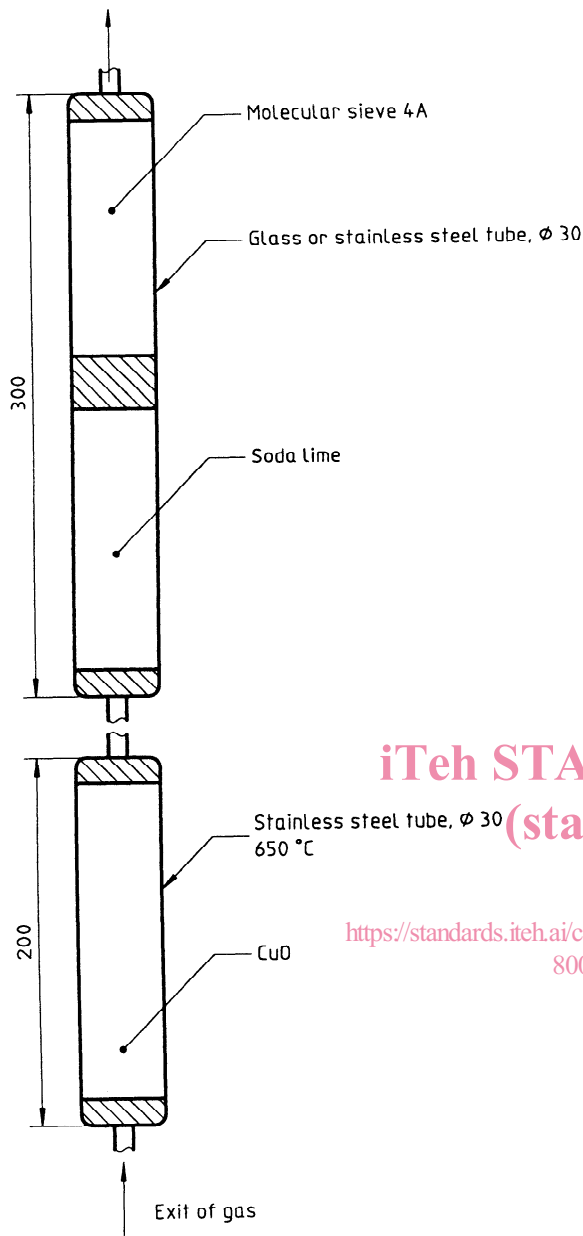


Figure 3 — Oxygen purification system

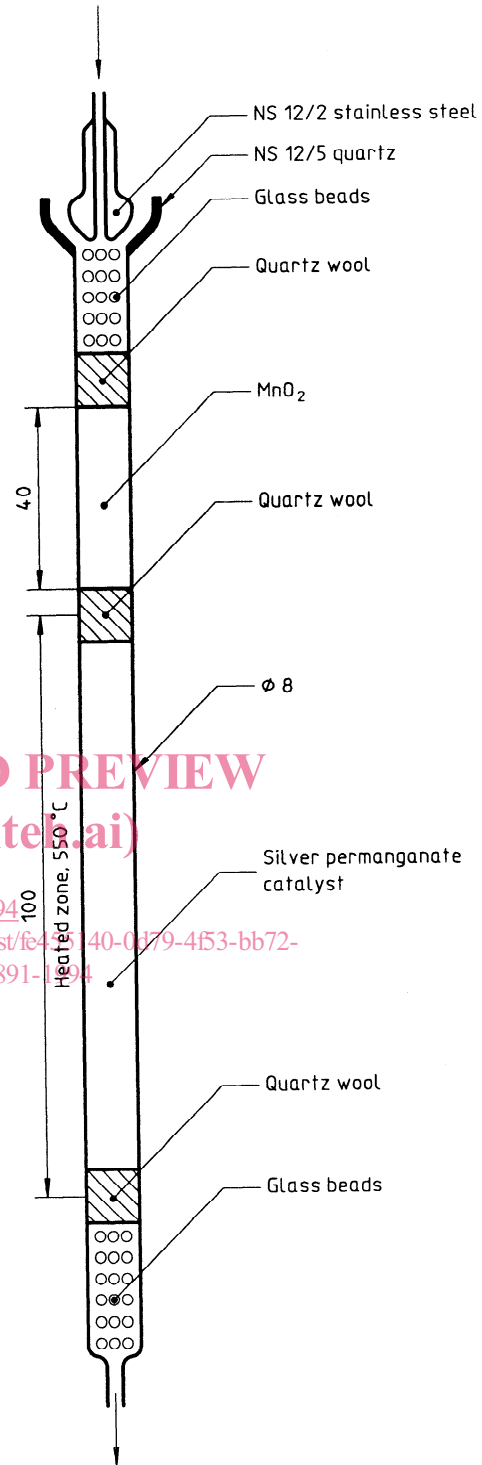


Figure 4 — Combustion gas purification system

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Dimensions in millimetres

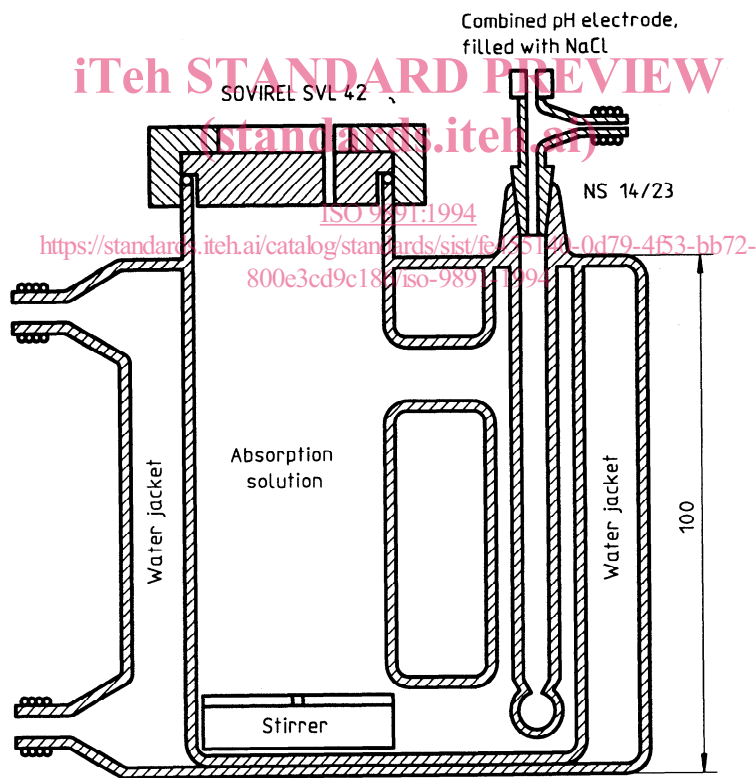
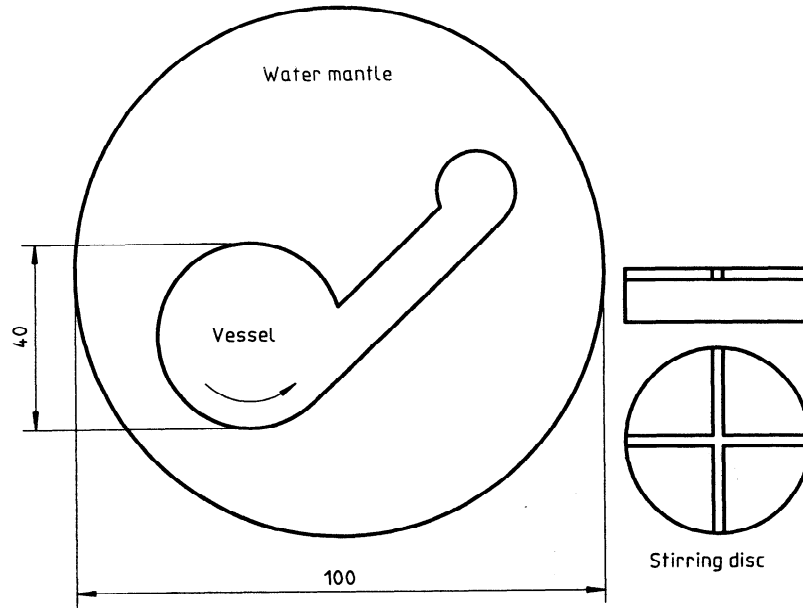


Figure 5 — Titration vessel