SLOVENSKI STANDARD
SIST ISO 182-4:1996

01-junij-1996

Polimerni materiali - Določanje tendence zmesi in proizvodov na osnovi homo- in kopolimerov vinilklorida, da pri povišanih temperaturah sproščajo klorovodik ali druge kisle produkte - 4. del: Potenciometrična metoda

Plastics -- Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures -- Part 4: Potentiometric method

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Plastiques -- Détermination de la tendance des compositions à base d'homopolymères et copolymères de chlorure de vinyle à dégager du chlorure d'hydrogène et éventuellement d'autres produits acides à températures élevées -- Partie 4: Méthode potentiométrique

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Ta slovenski standard je istoveten z: **ISO 182-4:1993**

ICS:

83.080.20	Plastomeri	Thermoplastic materials

SIST ISO 182-4:1996 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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INTERNATIONAL STANDARD

ISO 182-4

First edition
1993-04-01

Plastics — Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures —

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Part 4: Potentiometric method

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Plastiques — Détermination de la tendance des compositions à base d'homopolymères et copolymères du chlorure de vinyle à dégager du chlorure d'hydrogène et éventuellement d'autres produits acides à températures élevées —

Partie 4: Méthode potentiométrique

Reference number
ISO 182-4:1993(E)

SIST ISO 182-4:1996

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.

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.

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International Standard ISO 182-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 6, *Ageing, chemical and environmental resistance*.

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Together with the three other parts of ISO 182, it cancels and replaces ISO Recommendation R 182:1970, of which the four parts of ISO 182 constitute a technical revision.

ISO 182 consists of the following parts, under the general title *Plastics — Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures*:

- *Part 1: Congo red method*
- *Part 2: pH method*
- *Part 3: Conductometric method*
- *Part 4: Potentiometric method*

Annexes A and B of this part of ISO 182 are for information only.

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Part 4:
Potentiometric method

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WARNING — The use of this part of ISO 182 may involve hazardous materials, operations and equipment. This part of ISO 182 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 182 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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1 Scope

1.1 This part of ISO 182 specifies a method for the determination of the thermal stability at elevated temperature of compounds and products based on vinyl chloride homopolymers and copolymers (in the following text abbreviated as PVC) which undergo dehydrochlorination (the evolution of hydrogen chloride).

1.2 The method may be used as a quality control test during manufacture and conversion of PVC compounds. It may also be used for the characterization of PVC compounds and products, especially with regard to the effectiveness of their heat-stabilizing systems.

It is suitable for coloured PVC compounds and products for which a discolouration test under the action of heat may be unsatisfactory.

1.3 The method is recommended for compounded PVC materials and products only, although it can be used for polymers in powder form under appropriate conditions to be agreed upon between the interested parties. The method is not recommended for PVC compounds in the form of dry blends, since such materials may not be sufficiently homogeneous.

1.4 PVC compounds and products may evolve other decomposition products in addition to hydrogen chloride at elevated temperatures. A limited number of these products, originating from the decomposition of certain comonomers (such as vinyl esters of organic acids) or of plasticizers, stabilizers and other additives, may effect the pH or the conductivity of an aqueous solution when they are absorbed. Consequently, the results obtained for different products by the methods described in Parts 2 and 3 of ISO 182 may not be comparable with those obtained using the method described in the present part of ISO 182.

1.5 This part of ISO 182 specifies a potentiometric method for the determination of chloride ion (Cl^-) concentration (expressed as pCl) in an absorbing solution, independent of the presence of other ions. The value pCl is defined as $-\lg c_{Cl}$, where c_{Cl} is the molar concentration of chloride ions. This method is, therefore, particularly recommended for plasticized PVC compounds and copolymers.

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1.6 This method may also be applied to other plastics materials that can evolve hydrogen chloride when heated under the conditions prescribed by the relevant specifications, or as agreed upon between the interested parties.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 182. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 182 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 182-2:1990, *Plastics — Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures — Part 2: pH method.*

ISO 182-3:1993, *Plastics — Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures — Part 3: Conductometric method.*

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

3 Definition

For the purposes of this part of ISO 182, the following definition applies.

3.1 stability time, t_s: Time, measured by reference to a predetermined change in the pCl of an absorbing solution, required for a certain amount of hydrogen chloride to be evolved when a prescribed mass of PVC compound or product is maintained at an elevated temperature under the test conditions specified in this part of ISO 182.

4 Principle

A test portion of the PVC compound or product is maintained at an agreed temperature in a nitrogen gas stream and the hydrogen chloride evolved is absorbed in a given amount of an appropriate solution. The amount of hydrogen chloride evolved is determined potentiometrically in relation to the recorded change in pCl of the absorbing solution.

5 Reagents

During the test, use only reagents of recognized analytical grade in accordance with ISO 6353-2.

5.1 Pure nitrogen, containing less than 6 ppm oxygen and less than 0,1 ppm carbon dioxide by volume. The purity shall be such that when the gas is passed through the absorbing solution for 1 h at a rate of 7,2 l/h ± 0,1 l/h, the conductivity of the water remains unchanged.

The gas shall be dried by passing it through a suitable drying agent and the flow-rate through the dehydrochlorination cell adjusted by means of a needle valve and measured using a suitable flowmeter.

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5.2 Hydrochloric acid, aqueous solution, c(HCl) = 0,01 mol/l.

5.3 Distilled or demineralized water.

5.4 Potassium nitrate (KNO_3)**, potassium sulfate** (K_2SO_4) or other salts, for the preparation of the absorbing solution (see 10.4).

6 Apparatus

The general arrangement of the apparatus, shown in figure 1, includes a re-usable dehydrochlorination cell A. This cell may be replaced by a disposable cell B.

6.1 Dehydrochlorination cells.

6.1.1 Cell A (re-usable), with shape and dimensions as shown in figure 2.

A recommended procedure for cleaning the cell is given in annex A.

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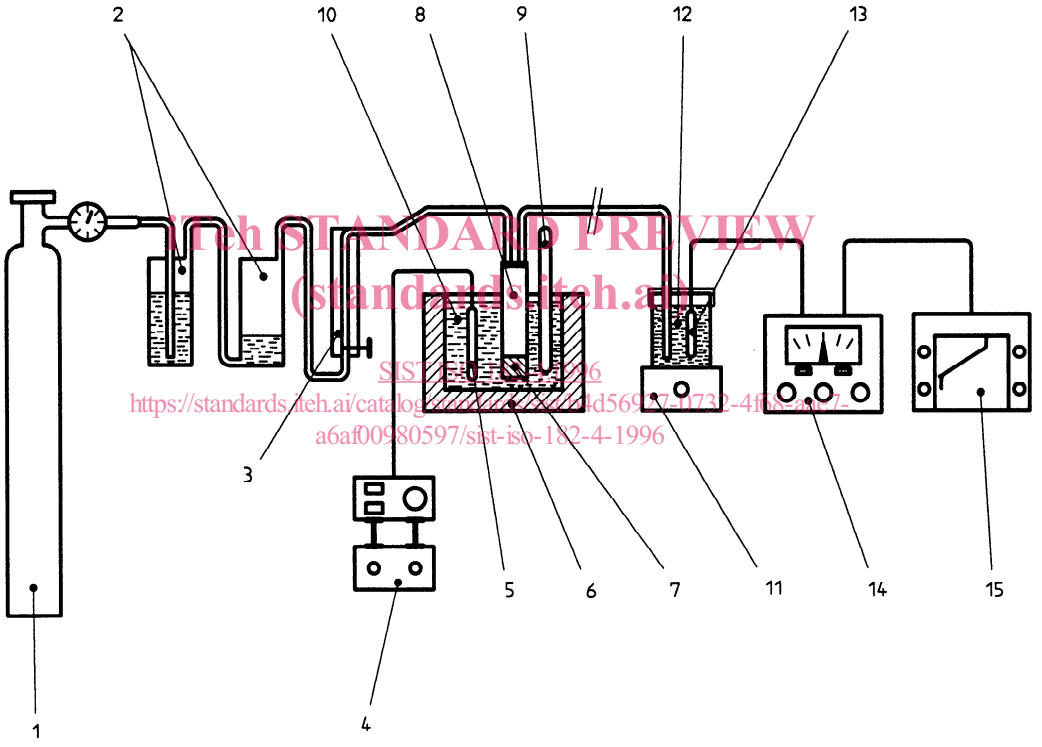
6.1.2 Cell B (disposable), with shape and dimensions as shown in figure 3.

NOTE 1 Other types of cell may be employed if it has been proved that the results obtained are equivalent to those obtained with one of the cells described in 6.1.1 and 6.1.2.

6.2 Test portion holder, for use with cell A. The test portion is supported on a porous sintered-glass disc (grade P 100, see ISO 4793), 10 mm in diameter.

To prevent blocking of the porous disc, it is advisable to place a thin, soft layer of glass wool between it and the test portion.



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1 N_2 cylinder
2 Purification train
3 Ball flowmeter
4 Electronic temperature controller (scale division 0,1 °C)
5 Temperature sensor
6 Heating bath
7 PVC test portion
8 Dehydrochlorination cell
9 Thermometer (scale division 0,1 °C)
10 Silicone oil
11 Magnetic stirrer
12 Absorbing solution
13 Measuring electrode(s)
14 Potentiometer
15 Recorder

Figure 1 — General arrangement of apparatus

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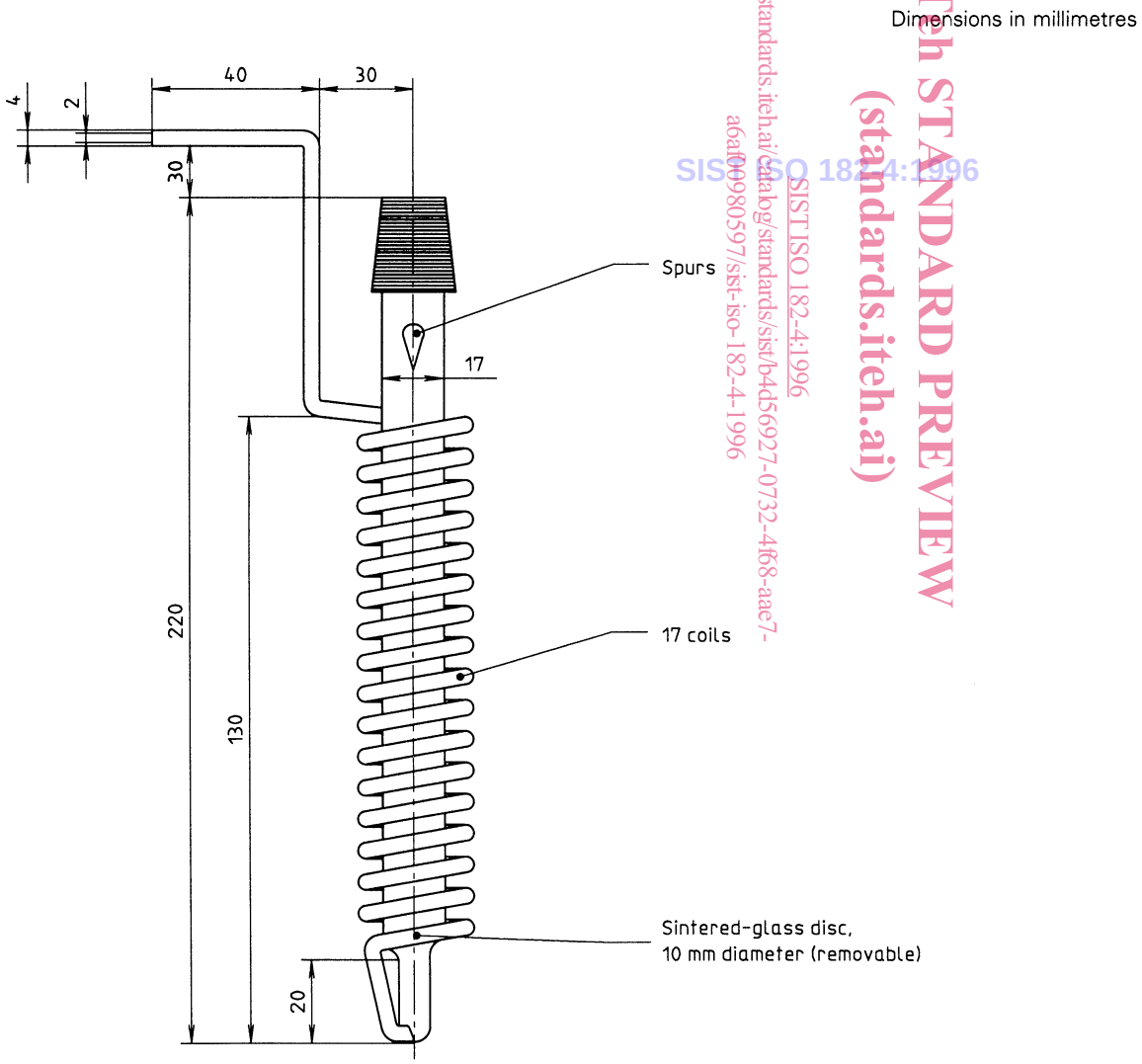

Figure 2 — Cell A (re-usable) for dehydrochlorination of PVC samples

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Dimensions in millimetres

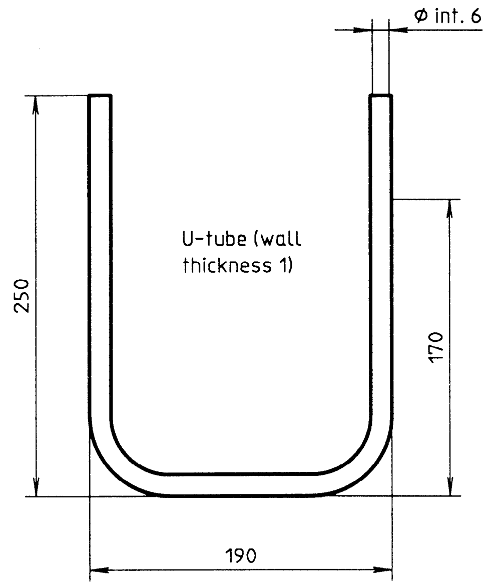

Figure 3 — Cell B (disposable) for dehydrochlorination of PVC samples

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A recommended procedure for cleaning the disc support is given in annex A.

6.3 Glass connection tube, for use with cell A, having dimensions as shown in figure 4. The connection tube is secured to cell A by two springs fixed to hooks on the ground-glass joints. The tube shall be provided with an insulating jacket.

6.4 Expansion joints and cell connections, for use with cell B. Cell B is connected to the apparatus through flexible polytetrafluoroethylene (PTFE) and silicone rubber tubes. Special joints allow for thermal expansion. The complete joint arrangement is shown in figure 5.

6.5 Oil bath, with a capacity of at least 10 l. The bath shall be capable of operating in the temperature range 170 °C to 210 °C and of maintaining the test temperature with an accuracy of 0,1 °C.

The bath shall be designed in such a way that the temperature distribution is even throughout, and shall have a thermal capacity sufficient to avoid temperature change when the dehydrochlorination cell is immersed in it.

6.6 Thermometer, with a scale suitable for reading the heating bath temperature in the range 170 °C to 210 °C and with a scale division of 0,1 °C.

6.7 Balance, with a scale division of 1 mg.

6.8 Measurement cell

A suitable measurement cell is shown in figure 6. If the diameters of the measurement electrode and the feed tube for the gases from decomposition of the test portion are sufficiently small, a 400 ml Erlenmeyer flask is a suitable alternative measurement cell.

A recommended procedure for cleaning the cell is given in annex A.

6.9 Magnetic stirrer, capable of providing gentle agitation within the measurement cell.

6.10 Potentiometer, for the determination of the pCl.

6.10.1 Specific electrode for the Cl^- ions, having a precision of at least 0,01 pCl. The sensitive element is a silver chloride (AgCl) crystal (see annex B).

6.10.2 Reference electrode on the basis of calomel (mercurous chloride) (Hg/Hg_2Cl_2/saturated KCl) or of mercurous sulfate (Hg/Hg_2SO_4/saturated K_2SO_4) or other, according to the specifications of the supplier of the specific electrode.

6.10.3 Extension (salt bridge), for the reference electrode. For the measurement of Cl^- ions, a salt bridge is absolutely necessary if a mercurous chloride electrode is used.

NOTE 2 Some "combined" specific electrodes are commercially available which also include a reference electrode (see annex B).

6.10.4 Electronic millivoltmeter.

The apparatus shall be provided with a device for automatic temperature compensation and equipped with an output for a recording device.

6.11 Stopclock, or other suitable timing device, if not included in the recorder.

6.12 Flowmeter, for example a rotameter, or other suitable device capable of measuring a gas flow-rate within the range 120 cm³/min ± 4 cm³/min.

7 Preparation of test samples

The measured stability times t_s depend to some extent on the surface area of the prepared test portions as well as on their thermal history. Any cutting or grinding of a material necessary to produce the test portions shall be conducted in a uniform manner, avoiding heating of the material.

NOTE 3 Cryogenic grinding is recommended.