# FINAL DRAFT

# INTERNATIONAL STANDARD

# ISO/FDIS 182-3

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ATS on: ates on: Teh STA temperatures — 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

# (staPart 3:ds.iteh.ai) Conductometric method

https://standards.itch.ai/cPlastiques — Détermination de la tendance des compositions et 88 produits à base d'homopolymères et de copolymères du chlorure de vinyle à dégager du chlorure d'hydrogène et éventuellement d'autres produits acides à températures élevées —

Partie 3: Méthode conductimétrique

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ISO/FDIS 182-3

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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 document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 182-3:1993), which has been technically revised.

The main changes are as follows:

- adding the use of alternative heating baths such as metal blocks.

A list of all parts in the ISO 182 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

# 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

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

# 1 Scope

**1.1** This document 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** This document is applicable to the characterization of PVC compounds and products, especially with regard to the effectiveness of their heat-stabilizing systems.

It is applicable to coloured PVC compounds and products where a discolouration test under the action of heat may be unsatisfactory.

**1.3** This document is applicable to compounded PVC materials and products. It can be applicable to polymers in powder form under appropriate conditions, to be agreed upon between the interested parties.

This document does not apply to PVC compounds in the form of dry blends, since such materials can be not sufficiently homogeneous.

**1.4** This document does not apply to PVC compounds and products which evolve other decomposition products, in addition to hydrogen chloride, at elevated temperatures that can affect the conductivity of water when they are absorbed into it. In this case, a method suitable for the determination of chloride ion (Cl<sup>-</sup>) in the absorbing solution shall be used (see ISO 182-4<sup>[2]</sup>).

**1.5** This document can also be applied to other plastics materials which can evolve hydrogen chloride or other hydrogen halides when heated under the conditions prescribed by the relevant specifications, or as agreed upon between the interested parties.

# 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 4793, Laboratory sintered (fritted) filters — Porosity grading, classification and designation ISO 6353-2, Reagents for chemical analysis — Part 2: Specifications — First series

# 3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

## 3.1

## stability time

#### ts

time, measured by reference to a predetermined change in the conductivity of absorbing demineralized water, 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 document

# 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 demineralized water. The amount of hydrogen chloride evolved is determined in relation to the recorded change in conductivity of the water.

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# 5 Reagents https://standards.iteh.ai/catalog/standards/sist/210cc5e1-6366-4b82-bb83-

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 demineralized water (see 5.2) for 1 h at a rate of  $(7,2 \pm 0,1)$  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.

**5.2 Demineralized water,** with a conductivity not higher than 5 μS/cm.

# **6** Apparatus

The general arrangement of the apparatus is shown in <u>Figure 1</u>.

## 6.1 Dehydrochlorination cells.

**6.1.1 Glass Cell A** (re-usable), with shape and dimensions as shown in <u>Figure 2</u>.

A recommended procedure for cleaning the cell is given in <u>Annex A</u>.

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

NOTE Other types of cell can 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 specified in 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.

A recommended procedure for cleaning the disc support is given in <u>Annex A</u>.



#### Figure 1 — General arrangement of apparatus

Dimensions in millimetres



#### Key

- 1 spurs
- 2 17 coils
- 3 sintered-glass disc, 10 mm diameter (removable)

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

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**Dimensions in millimetres** 



1

2 disposable nitrogen inlet tube

3 sample

Key

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

6.3 Glass connection tube, for use with cell A, connecting the dehydrochlorination cell to the measurement cell and 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.



Key 1,2 spurs

# Teh STANDARD PREVIEW

# Figure 4 — Glass tubes for connecting the dehydrochlorination cell to the measurement cell (use for cell A)

6.4 Heating bath (e.g. oil bath)/Heating block (e.g. metal block) including temperature controller with a capacity of at least 10 l. The bath / block shall be capable of operating in the temperature range 170 °C to 210 °C and of maintaining the test temperature with an accuracy of  $\pm 0.1$  °C.

The bath/block shall be designed so 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.

**6.5** Thermometer, with a scale suitable for reading the heating bath/heating block temperature in the range 170 °C to 210 °C and with a resolution of 0,1 °C.

**6.6 Balance**, with a resolution of 1 mg.

## 6.7 Measurement cell.

In general, the measurement cell is a glass vessel with lid and with the relevant openings for the measurement electrode and the feed tube and it shall look like in <u>Figure 1</u>. If the diameters of the measurement electrode and the feed tube for the gases from decomposition of the test portion are sufficiently small, a 300 ml Erlenmeyer flask is a suitable alternative measurement cell.

A recommended procedure for cleaning the cell is given in <u>Annex A</u>.

**6.8 Magnetic stirrer,** capable of providing gentle agitation within the measurement cell. Only necessary in case using an oil bath.

**6.9 Conductance meter,** with a conductivity probe constant of 1,0 cm<sup>-1</sup> to 1,2 cm<sup>-1</sup>, giving direct readings in  $\mu$ S/cm, and preferably equipped with an automatic temperature compensating device.

The cell constants should be determined by means of a specified standard solution, e.g. a conductivity standard of 100  $\mu$ S/cm, because in general electrodes are not always stable.

NOTE Graphite electrodes have been shown to operate in a more trouble-free manner than platinized platinum electrodes.

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

**6.11 Flowmeter,** for example a rotameter, or other suitable device capable of measuring a gas flow-rate within the range  $(7,2 \pm 0,1)$  l/h.

## 7 Preparation of test samples

#### 7.1 General

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.

Cryogenic grinding is recommended.

## 7.2 PVC plastisols

Spread these materials on glass plates and gel them in an oven at an agreed temperature so that sheets 0,5 mm thick are formed. Cut these sheets into squares approximately 2 mm on a side.

7.3 PVC pellets, extrudates, mouldings, thick sheet, etc. 1-6366-4b82-bb83-

Cut or grind these materials so that more than 80 % of the material passes through a 2 mm sieve in accordance with R 20/3 series, specified in ISO 565, and an amount adequate to prepare the test portions is retained on a 1,4 mm sieve.

## 7.4 PVC film and sheet

Cut these materials into squares or cubes having sides no longer than 2 mm.

## 7.5 PVC coatings

Separate these materials from the substrate and then treat as in  $\underline{7.2}$  or  $\underline{7.3}$ .

#### 7.6 Cable and conductor insulation or sheating

Cut thin slivers of the dimensions indicated in 7.2.

## 8 Number of tests

Conduct at least two determinations on each sample.

## 9 Temperatures for dehydrochlorination

The test temperatures are preferably:

200 °C for unplasticized compounds and products;