INTERNATIONAL STANDARD

ISO 182-2

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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

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 2: Méthode au pH

Reference number
ISO 182-2:1990(E)

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.

International Standard ISO 182-2 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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 methods*
- *Part 2: pH method*
- *Part 3: Conductometric method*
- *Part 4: Potentiometric method*

Annex A of this part of ISO 182 is for information only.

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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

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.

1 Scope

1.1 This part of ISO 182 specifies a method for the determination of the thermal stability at elevated temperatures of compounds and products based on vinyl chloride homopolymers and copolymers (in the following text abbreviated to 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 be used also for the characterization of PVC compounds and products, especially with regard to the effectiveness of their heat-stabilizing systems.

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

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

PVC compounds and products may evolve decomposition products in addition to hydrogen chloride at elevated temperatures. A limited number of these decomposition products may affect the pH of an absorbing solution. It is not possible to compensate for this effect within the scope of this part of ISO 182, and therefore care is necessary in comparing results for dissimilar compounds and products. In this case, a method suitable for the determination of the chloride ions in the absorbing solution shall be used (see ISO 182-4).

1.4 The method may 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 when 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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

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

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.

stability time, t_s: The time, measured by reference to a predetermined change in the pH of an absorbing solution, required for a certain amount of hydrogen chloride to be evolved when a given mass of PVC compound or product is maintained at an elevated temperature under the conditions of test described 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 gas stream and the hydrogen chloride evolved is absorbed in a 0,1 mol/l solution of sodium chloride. The amount of hydrogen chloride evolved is measured by recording the change in pH of the sodium chloride solution.

5 Reagents and materials

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

5.1 Supply of pure nitrogen, containing less than 6 ppm of oxygen and less than 0,1 ppm of carbon dioxide by volume. The purity shall be such that, when the gas is passed through a 0,1 mol/l solution of sodium chloride at pH 6 for 1 h at a rate of 7,2 l/h ± 0,1 l/h, the pH of the solution remains in the range 5,7 to 6,7. The gas shall be dried by passing it through a suitable drying agent, and the flow rate adjusted by means of a needle valve and measured using a suitable flowmeter.

5.2 Sodium chloride solution, $c(\text{NaCl}) = 0{,}1$ mol/l.

Dissolve 5,84 g ± 0,01 g of sodium chloride in grade 3 distilled water as defined in ISO 3696, which has been boiled to remove carbon dioxide. Dilute to 1 litre with boiled distilled water of the same grade.

5.3 Sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l.

5.4 Hydrochloric acid, $c(\text{HCl}) = 0{,}1$ mol/l.

6 Apparatus

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

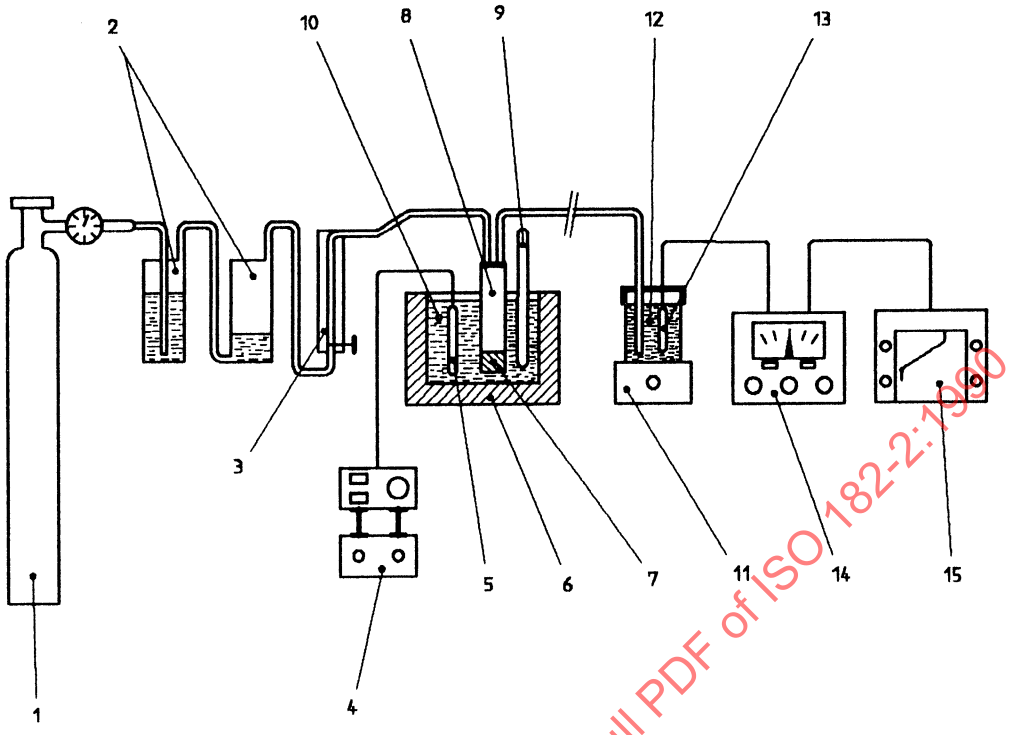
6.1 Dehydrochlorination cells

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

A recommended procedure for cleaning is given in annex A.

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

6.1.3 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.



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 pH-meter
15 Recorder

Figure 1 — General arrangement of apparatus

Dimensions in millimetres

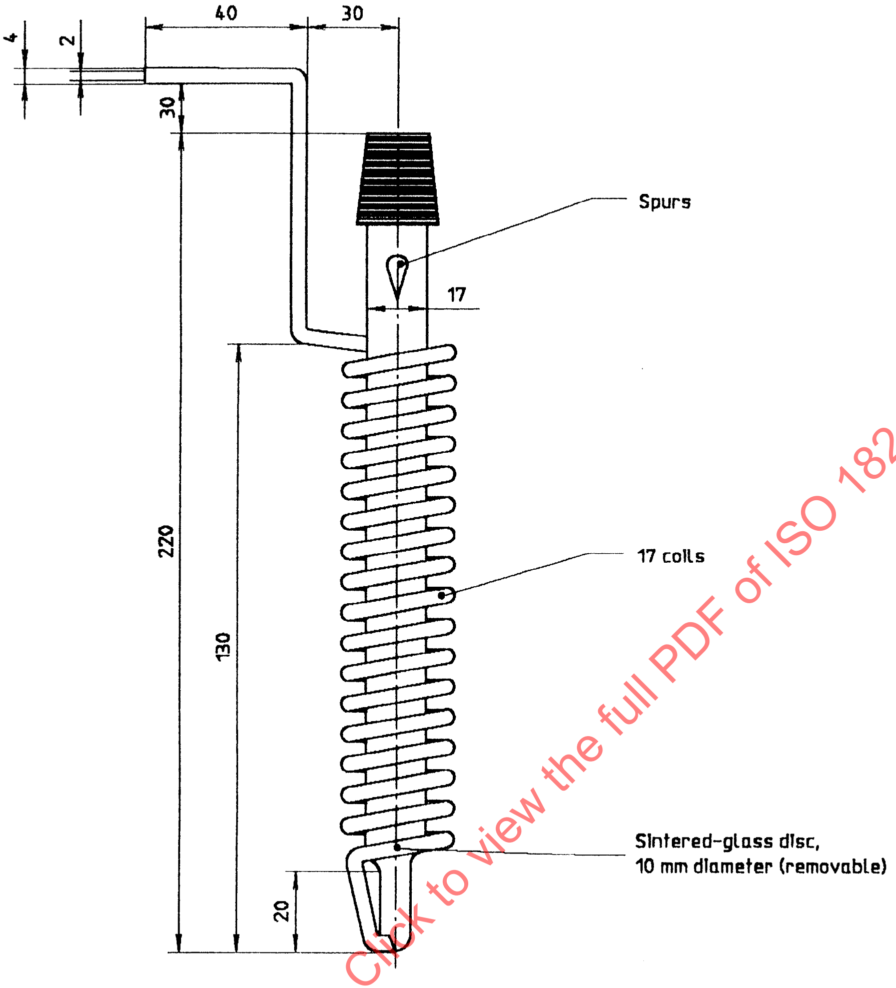


Figure 2 — Cell A (re-usable) for dehydrochlorination of PVC samples

Dimensions in millimetres

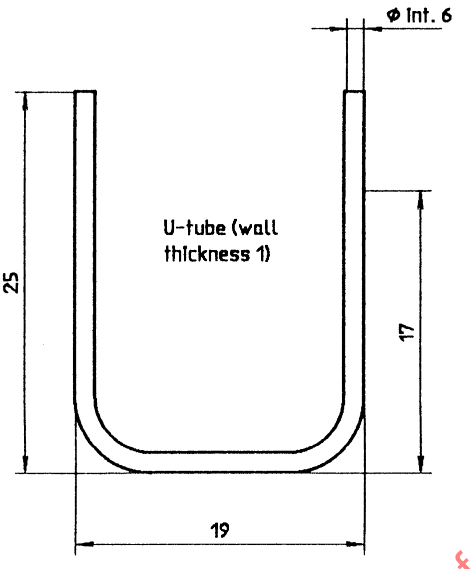


Figure 3 — Cell B (disposable) for dehydrochlorination of PVC samples

6.2 Test-portion holder, for use with cell A.

The test portion is supported on a porous sintered-glass disc (P 100, see ISO 4793), 10 mm in diameter.

To avoid the porous disc becoming blocked, it is advisable to place a thin layer of glass wool between it and the test portion.

A recommended procedure for cleaning is given in annex A.

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

6.4 Expansion joints and cell connections, for use with cell B.

Cell B is connected to the apparatus by flexible PTFE and silicone rubber tubes. Special joints are provided to allow for thermal expansion. The complete arrangement is shown in figure 5.

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

The bath shall be designed so that the temperature distribution is even throughout, and have a thermal capacity sufficent to avoid the temperature changing when the cell is immersed.

6.6 Thermometer, with a suitable scale for reading the 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. Where the diameters of the measurement electrode and the feed tube for the gases from decomposition of the test portion are sufficiently small, a 200 ml conical flask is a suitable alternative measurement cell.

A recommended procedure for cleaning is given in annex A.

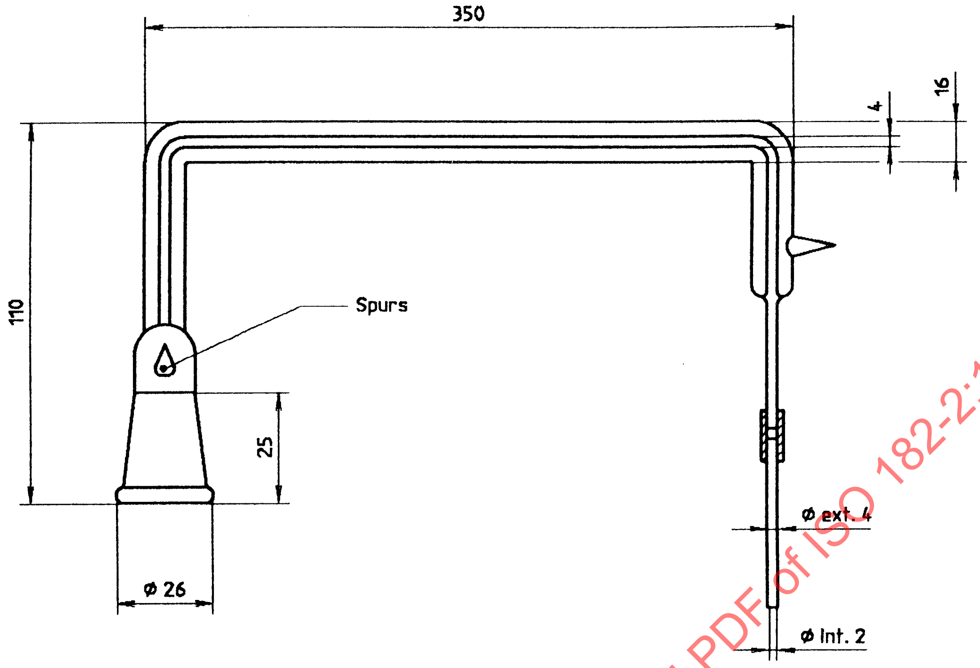
6.9 Magnetic stirrer, capable of providing gentle agitation within the measurement cell.

6.10 pH-meter with suitable electrodes, accurate to at least 0,01 pH units.

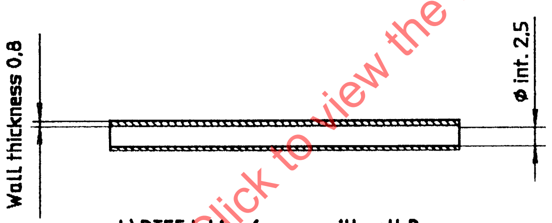
The pH-meter should preferably be provided with a device for automatic temperature compensation and equipped with an output for a chart recorder.

The apparatus shall be calibrated with buffer solutions covering the appropriate pH range.

Dimensions in millimetres



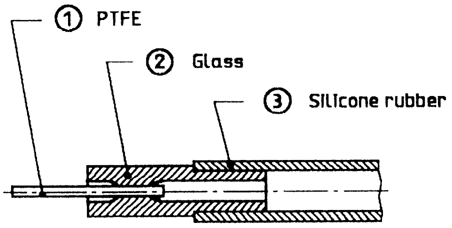
a) Glass tube for use with cell A



b) PTFE tubing for use with cell B

Figure 4 — Tubes for connecting dehydrochlorination cell to measurement cell

Dimensions in millimetres



1 PTFE tube: ⌀ ext. 4,1
⌀ int. 2,5
2 Glass tube: ⌀ ext. 8
⌀ int. 6
3 Silicone rubber tube: ⌀ ext. 12
⌀ int. 6

Figure 5 — Expansion joint for connection on each side of cell B

Dimensions in millimetres

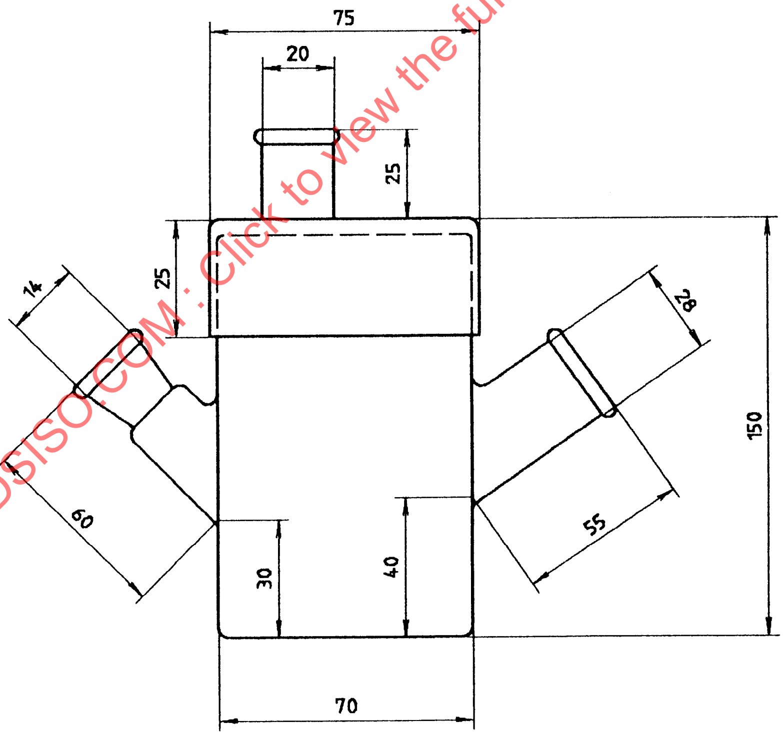


Figure 6 — Example of a suitable measurement cell

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

6.12 Flowmeter, Rotameter or other suitable flowmeter capable of measuring a gas flow rate within the range 120 cm^3/min ± 4 cm^3/min.

7 Preparation of test samples

The measured stability times 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 carried out in a uniform manner. Heating of the material during grinding shall be avoided.

7.1 PVC plastisols

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

7.2 PVC pellets, extrudates, mouldings, thick sheet, etc.

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

7.3 PVC film and sheet

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

7.4 PVC coatings

Separate these materials from the substrate and then treat as in 7.2 or 7.3.

7.5 Insulation or sheathing of cables and conductors

Cut thin slivers with the dimensions indicated in 7.2.

8 Number of tests

Conduct at least two determinations on each sample.

9 Temperatures for dehydrochlorination

These are preferably:

200 °C for unplasticized compounds and products;

200 °C for compounds and products for cable insulation and sheathing;

180 °C for plasticized compounds and products.

10 Test procedure

10.1 Preparation of test portion

Weigh, to the nearest 0,01 g, 2 g of the test sample, prepared in accordance with the appropriate part of clause 7.

Introduce this test portion into the chosen dehydrochlorination cell (6.1) and connect the cell to the remainder of the apparatus, taking particular care to ensure that all joints are tightly closed and, where appropriate, that all ground-glass joints are sealed with a suitable stopcock grease.

10.2 Preliminary operations

Heat the oil bath (6.5) to the agreed test temperature. Start the flow of nitrogen, adjust to a flow rate of 120 cm^3/min ± 4 cm^3/min and keep it flowing for about 5 min to eliminate air. During this period, the dehydrochlorination cell shall not be heated. The nitrogen flowing through the cell shall be allowed to escape to the atmosphere without passing through the absorbing solution contained in the measurement cell (6.8).

10.3 Special precautions when using dehydrochlorination cell A

When using cell A (6.1.1), all the spirals of the preheating tube shall be completely immersed in the oil bath. The end of the gas-outlet tube shall be positioned about 5 mm from the bottom of the measurement cell.

10.4 Preparation of the measurement cell

Introduce 180 ml of sodium chloride solution (5.2) into the measurement cell and start the stirrer (6.9). Allow the measurement cell to reach temperature equilibrium in the range 23 °C ± 2 °C and maintain it at that temperature. Adjust the pH of the solution to 6,0 by the drop-by-drop addition of sodium hydroxide solution (5.3) or hydrochloric acid (5.4). Insert the pH-meter electrodes (see 6.10) into the solution to the depth recommended by the electrode manufacturer.

NOTE 1 It may be necessary to conduct the test in a temperature-controlled room. Alternatively, it may be more convenient to surround the measurement cell with a water bath in order to maintain the temperature at 23 °C ± 2 °C.

10.5 Decomposition of the test portion

Immerse the dehydrochlorination cell rapidly in the oil bath and immediately place the end of the connecting tube into the sodium chloride solution contained in the measurement cell.

Start the stopclock (6.11) and/or the recorder at the moment when the tube is immersed in the sodium chloride solution, and continue the test at least until the pH value decreases to 3,8.

11 Expression of results

Record the time, in minutes, taken to reach the pH value of 3,8 as the stability time t_s.

12 Precision

12.1 Repeatability

In a series of tests with cell A, the data shown in table 1 were obtained.

12.2 Reproducibility

In tests conducted with cell A on one product and in two laboratories over a period of 2 years, the data shown in table 2 were obtained.

In addition, a single product was tested in three laboratories using cell A. The data shown in table 3 were obtained.

Table 1 — Repeated determinations of stability time

Product	Type	Laboratory	Temperature of test °C	Stability time min		Number of tests
				Mean	Range	
PVC A	Rigid	1	200	93,6	91,3 to 94,7	5
PVC B	Rigid	1	200	106,1	103,6 to 108,1	5
PVC C	Plasticized	1	180	221,8	218,9 to 223,3	3

Table 2 — Repeated determinations of stability time for a single product over a 2 year period

Product	Type	Year	Laboratory	Temperature of test °C	Stability time min		Number of tests
					Mean	Range	
PVC D	Rigid	1	2 and 3	200	97	93 to 100	17
PVC D	Rigid	2	2 and 3	200	98	96 to 99	5

Table 3 — Repeated determinations of stability time in several laboratories using a single product

Product	Type	Laboratory	Temperature of test °C	Stability time min		
				Individual values	Mean	Range
PVC E	Rigid	4	200	89; 89	95	89 to 100
PVC E	Rigid	5	200	97; 97		
PVC E	Rigid	6	200	98; 100		

13 Test report

The test report shall include the following information:

a) a reference to this part of ISO 182;

b) the nature, form and designation of the PVC compound or product tested;

c) if appropriate, the manufacturer's name, where sampled and the degree of comminution of the test portion;

d) the type of dehydrochlorination cell used (cell A, cell B or other);

e) the test temperature;

f) the stability time t_s, in minutes, to the nearest half-minute (include individual values and the arithmetic mean);

g) any deviation from the procedure specified in this part of ISO 182;

h) the date of the test.