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Plastics — Intumescence properties of PVC materials and products — Test method for the measurement of expansion with the cone calorimeter

Plastiques — Propriétés d'intumescence des matériaux et produits en PVC — Méthode d’essai pour mesurer l’expansion à l’aide d'un calorimètre à cône

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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 www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 4, *Burning behaviour.*

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 www.iso.org/members.html.

Introduction

Polyvinyl chloride (PVC) is an example of an intumescent material that leaves much of the original carbon content as a solid residue. The presence of chlorine in PVC exerts its influence in two ways, causing an increase in:

— hydrogenated char formation (meaning that less flammable decomposition products are formed);

— generating HCl, which then acts as a gas phase scavenger slowing down further reactions of flammable products in the gas phase. [- CH_2 – CHCl- → - CH = CH - + HCl]

The expansion formed has two positive effects regarding fire safety:

— it is a barrier between the source of heat and the unaffected polymer material leading to a reduction of its rate of decomposition;

— it reduces the release of flammable gas.

ISO/TR 20118 provides information on fire properties of PVC materials.

The intumescent properties of PVC materials and products are likely, however, to be affected by their exact formulation and by the use of specific chemical species (e.g. intumescent chemicals), as processing aids or other components of the specific material. The aim of this document is to propose a test method to characterize the intumescence properties of PVC materials and products, by using the cone calorimeter heater as defined in standards ISO 5660-1, ISO 13927 and ISO 17554.

In this test method, the expansion of PVC materials and products is determined by the measurement of the height increase of a specimen when exposed to the radiated heat produced by an electrical conical heater. Intumescence is a property given to, or pre-existing in, some materials and used in fire safety as a way to increase fire performance and protect materials or products with regard to either reaction-to-fire or fire resistance.

Plastics — Intumescence properties of PVC materials and products — Test method for the measurement of expansion with the cone calorimeter

1 Scope

This document specifies a test method for assessing the expansion of PVC materials and products during their combustion under the effect of heat radiation.

This test method is also applicable to materials and products made from blends and mixtures of PVC with other polymers, such as PVC blended with a copolymer of styrene and acrylonitrile (PVC-SAN).

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 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 1043-1, *Plastics — Symbols and abbreviated terms — Part 1: Basic polymers and their special characteristics*

ISO 1043-4, *Plastics — Symbols and abbreviated terms — Part 4: Flame retardants*

ISO 3126, *Plastics piping systems — Plastics components — Determination of dimensions*

ISO 5660-1, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)*

ISO 13927, *Plastics — Simple heat release test using a conical radiant heater and a thermopile detector*

ISO 13943, *Fire safety — Vocabulary*

ISO 14934-2, *Fire tests — Calibration and use of heat flux meters — Part 2: Primary calibration methods*

ISO 14934-3, *Fire tests — Calibration and use of heat flux meters — Part 3: Secondary calibration method*

ISO 17554, *Reaction to fire tests — Mass loss measurement*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1043-1, ISO 1043-4, ISO 13943 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
intumescence
formation of a swelling/foaming substance in response to heat that insulates the underlying substrate

Note 1 to entry: Definition is adapted from ISO 13943.

Note 2 to entry: Intumescence is used to achieve passive fire protection for such applications as firestopping, fireproofing, gasketing and window casings.

[SOURCE: ISO 13943:2023, 3.268, modified — Note to entry has been added.]

3.2
flame retardant
substance added, or a treatment applied, to a material in order to suppress or delay the appearance of a flame and/or reduce the flame spread rate

Note 1 to entry: Flame retardants are activated by the presence of an ignition source and are intended to prevent or slow the further development of ignition by a variety of different physical and chemical methods. Various species of flame retardants, including smoke suppressants, alone or in combination, can lead to consistent lowering of heat release, flame spread, ignitability, (by increasing the time to ignition or the minimum heat flux for ignition), or smoke release.

Note 2 to entry: ISO 1043-4 lists the various categories of flame retardants.

[SOURCE: ISO 13943:2023, 3.192, modified — Notes to entry have been replaced.]

4 Symbols

e_{im} average initial thickness of the specimen, mm

e_{of} overall final thickness of the specimen, mm

E_p expansion rate of the material or product, %

E_s expansion rate of the specimen, %

5 Principle

The test method detailed in this document consists in measuring the rate of expansion of a PVC test specimen during heating and/or combustion under the effect of radiative heat generated by an electrical cone calorimeter heater.

The method is based on the observation that when a PVC specimen is exposed horizontally to incident radiative heat, this generally results in an increase of its volume, due to intumescence.

Test specimens are exposed to heat under ambient air conditions, while being subjected to a predetermined external heat flux of 50 kW/m^2. Measurements of the thickness of the test specimen are made before and after the test.

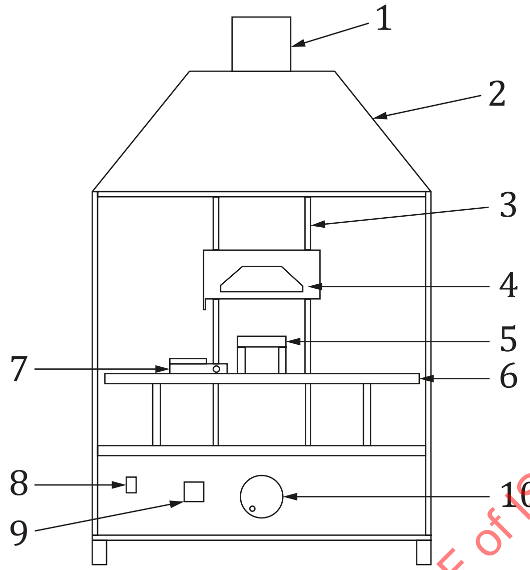
The expansion rate quantifies the intumescence property of the PVC test specimen and is calculated as the percentage of thickness increase before and after the test.

6 Test equipment

6.1 General

An example of schematic representation of test equipment is given in Figure 1. The individual components are described in detail in 6.2 to 6.9.

Test equipment conforming with either ISO 5660-1, ISO 17554 or ISO 13927, is suitable to conduct a test according to this document, although some of the measuring device components of the test equipment will not need to be used.



Key

1 extraction duct
2 hood
3 guiding column
4 cone-shaped radiant electric heater
5 specimen holder and support
6 support plate
7 carrousel
8 power switch
9 PID governor
10 handwheel

Figure 1 — Example of suitable test equipment

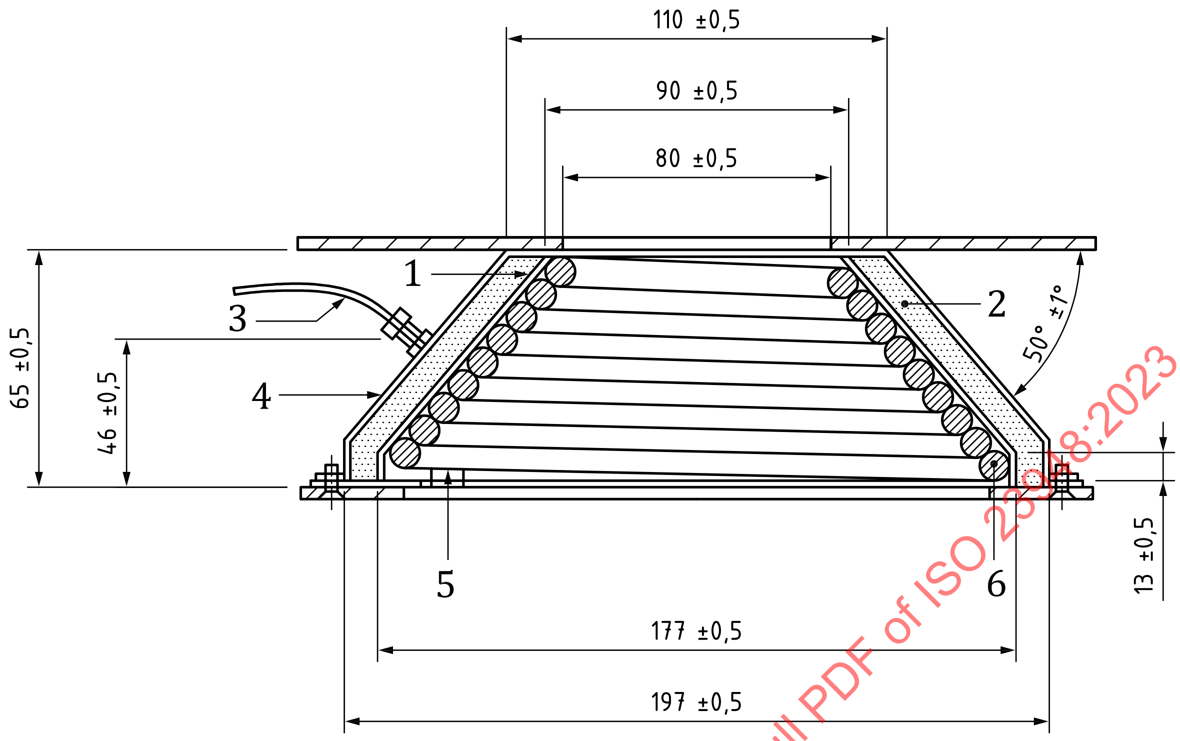
6.2 Cone-shaped radiant electrical heater

The active element of the heater shall consist of an electrical heater rod, capable of delivering 5 kW at the operating voltage, tightly wound into the shape of a truncated cone conforming with either ISO 5660-1, or ISO 17554, or ISO 13927, and highlighted as Figure 2. The heater shall be encased, from the outside, within a double-wall stainless steel cone, filled with a refractory fibre blanket of nominal thickness 6 mm and nominal density 100 kg m^{-3}.

The heat flux from the heater shall be maintained at a pre-set level by controlling the average temperature of three thermocouples (type K stainless steel sheathed thermocouples have proven suitable, but the use of other high-performance alloy materials is also acceptable), symmetrically disposed and in contact with, but not welded to, the heater element. Either 3,0 mm outside diameter sheathed thermocouples, with an exposed hot junction, or 1,0 mm to 1,6 mm outside diameter sheathed thermocouples, with an unexposed hot junction, shall be used.

The heater shall be capable of producing an incident heat flux on the surface of the specimen of up to at least 70 kW/m^2. The heat flux shall be uniform within the central 50 mm × 50 mm area of the exposed specimen surface, to within ±2 % for an incident heat flux level (at the centre) of 50 kW/m^2.

Dimensions in millimetres



Key

1 inner shell
2 refractory fibre packing
3 thermocouple (indicative position)
4 outer shell
5 space block
6 heating element

Figure 2 — Test equipment — Cone-shaped radiant electrical heater

6.3 Heat flux control

The heat flux control system shall be properly controlled so that it maintains the average temperature of the heater thermocouples during the calibration procedure described in 10.2 at the pre-set level, to within ±10 °C. Regulation of heat flux level shall be made by a proportional–integral–derivative controller (PID governor), based on the heater temperature.

6.4 Specimen holder

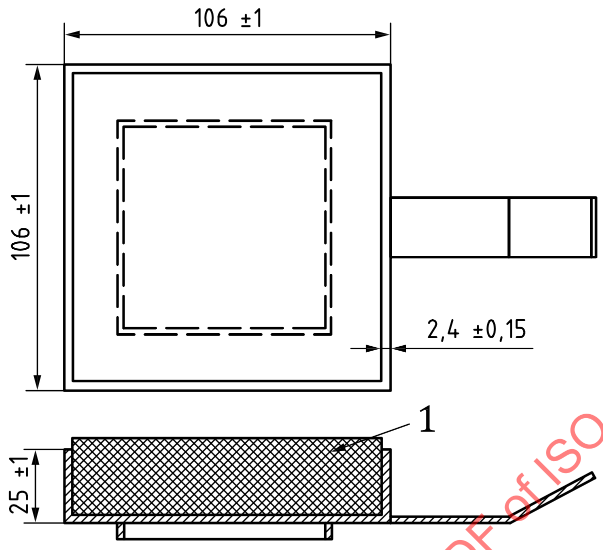
The specimen holder is shown in Figure 3. The specimen holder shall have the shape of a square pan with an opening of (106 ± 1) mm x (106 ± 1) mm at the top, and a depth of (25 ± 1) mm. The holder shall be constructed of stainless steel with a thickness of (2,4 ± 0,15) mm. It shall include a handle to facilitate insertion and removal, and a mechanism to ensure central location of the specimen under the heater.

The bottom of the holder shall be lined with a calcium silicate board with a density of 1 400 kg/m^3 and a thickness of (25,0 ± 0,5) mm.

The calcium silicate substrate shall be covered with aluminium foil (nominal thickness: 30 µm) before the test. The specimen shall be placed directly above the aluminium foil.

The distance between the bottom surface of the cone heater and the top surface of the specimen shall be adjusted to be (30 ± 1) mm. A steel gauge of 27 mm shall be used to fix the distance between the heater and the specimen surface prior to each test series.

Dimensions in millimetres



Key

1 calcium silicate substrate

Figure 3 — Specimen holder

6.5 Heat flux meter

The heat flux meter shall be of the Schmidt-Boelter (thermopile) type, with a design range of (10 to 100) kW/m^2. The target receiving the heat shall be flat, circular, of approximately 12,5 mm in diameter. It shall be coated with a durable matt black finish of surface emissivity ε = 0,95 ± 0,05. The body of the heat flux meter shall be water-cooled. A cooling temperature with the potential to cause condensation of water on the target surface of the heat flux meter shall not be used. Radiation shall not pass through any window before reaching the target. The heat flux instrument shall be robust, simple to set up and use, and stable in calibration. The instrument shall have a precision of ±3 % and a repeatability to within ±0,5 %.

The heat flux meter shall be positioned at a location equivalent to the centre of the specimen face during the calibration.

6.6 Fume hood and exhaust duct

The test equipment shall be equipped with an adequate extraction system consisting of a fume hood and an exhaust duct/fan operating during the entire test sequence. The extraction system shall provide sufficient flow for suitable extraction of all the smoke and combustion products, to ensure appropriate protection of the operator. By the nature of the materials or products being tested, the resistance to corrosion of the extraction system is one of the major criteria to be considered to ensure long-term efficiency and safe performance.

The rate of extraction shall not provide any air movements that deviate from the normal air movement around the test specimen during the test. The extraction system shall also limit soot re-deposition.

6.7 Dimensional measuring devices

The measuring devices shall conform to ISO 3126 and shall be capable of determining the specified dimensions with 1 mm minimum accuracy.

7 Suitability of a product for testing

The method is applicable to flat test specimens, that shall be permitted to be homogeneous, multi-layered, or coated.

The test method is limited to test specimens of a maximum initial thickness of 10 mm.

8 Preparation of the test specimen

8.1 Test specimens

8.1.1 Unless otherwise specified, five test specimens shall be tested for each different exposed surface.

NOTE Additional test specimens might be needed to determine optimal exposure duration (see 11.4).

8.1.2 The test specimens shall be representative of the product and shall be cut out or machined from the product to a square plate with sides measuring 50 ± 1 mm. The maximum thickness allowed is 10 mm.

8.1.3 The test specimen shall have an essentially flat surface. For tubular PVC products, test specimens with an essentially flat surface shall be obtained after softening the material in an oven at a temperature in the range of 125 °C to 150 °C, followed by flattening, and by cutting out the excess or machining the sample to the appropriate dimensions. The user shall ensure that the softening temperature used does not affect the fire properties (particularly the propension for intumescence) of the product.

8.1.4 Products with a normal thickness of 10 mm or less shall be tested using their full thickness. For products with a normal thickness of greater than 50 mm, the specimens shall be obtained by cutting away the unexposed face to reduce the thickness to one of 10 mm or less (by cutting out the excess or machining the sample).

8.1.5 Asymmetrical products and products which contain layers of different materials shall be tested by exposing to the radiant heat the surface that will be exposed in use, as described in 8.1.4. If the surface to be exposed in use is not defined, a first test shall be performed on each side. If the results for both surfaces are identical, 3 more tests shall be performed, and the result shall be present as applicable to both surfaces. If the results for both surfaces differ, 4 more tests shall be performed for each surface. In that case, the results shall be presented separately for each surface.

8.2 Conditioning of specimens

PVC specimens are normally not sensitive to moisture. Test specimens shall be conditioned at room temperature, between 15 and 30 °C, for a minimum of one hour before testing.

For samples that are sensitive to moisture, test specimens shall be conditioned to constant mass at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) % in accordance with ISO 554. Constant mass shall be considered to be reached when two successive weighing operations, carried out at an interval of 24 hours, do not differ by more than 0,1 % of the mass of the specimen or 0,1 g, whichever is the greater.

9 Test environment

The apparatus shall be located in an essentially draught-free environment in an atmosphere of relative humidity between 20 % and 80 % and a temperature between 15 °C and 30 °C.

10 Calibration

10.1 Heat flux meter

The heat flux meter shall be calibrated at least once every two years in accordance with ISO 14934-2. If another calibration scheme is used, with 2 heat flux meters, the primary one shall be calibrated in accordance with ISO 14934-2 at least every 5 years, and the secondary heat flux meter shall be calibrated in accordance with ISO 14934-3 at least every 6 months. Details on requirements for heat flux meter calibrations are available in ISO 14934-1[3] and ISO 14934-4[4].

10.2 Conical heater

The cone heater shall be calibrated at least every six months, at 50 kW/m^2, using the heat flux meter, for a distance between the bottom surface of the cone heater and the surface of the target of the heat flux meter of 30 mm. The set-point temperature of the cone heater shall be noted for every test day. Any deviation of more than 10 °C from the initial set point shall be reported and the system shall be re-calibrated.

NOTE Day control using a working heat flux meter is an acceptable substitute to replace temperature set-point monitoring.

10.3 Timer

The timer shall be calibrated at least every 3 years. with a calibration accuracy of less than 1 s.

10.4 Dimensional measurement devices

All dimensional measurement devices to be used, such as a micrometre, shall be calibrated at least every 3 years, with a calibration accuracy of less than 5 % of the measured value.

11 Test procedure

11.1 General precautions

WARNING — So that suitable precautions are taken to safeguard the health of the operators, everyone associated with the fire tests shall be made aware of the possibility that toxic or harmful gases will potentially be evolved during the exposure of test specimens.

The test procedures used in this test method involve high temperatures and combustion processes. Therefore, the potential exists for operator burns or for the ignition of extraneous objects or clothing. The operator shall use protective gloves for insertion and removal of test specimens. Neither the cone heater nor the associated fixtures shall be touched while hot except with the use of protective gloves. The exhaust system of the apparatus shall be checked for proper operation before testing and shall discharge into a building exhaust system with adequate capacity. The possibility of the violent ejection of molten hot material or sharp fragments from some kinds of specimens when irradiated cannot totally be discounted and it is therefore essential that eye protection be worn.

11.2 Specimen thickness

The average initial thickness (e_{im}) of each test specimen shall be determined as the arithmetical mean of 3 measurements of the thickness, distributed over the surface of the test specimen. Each measurement shall be determined with an accuracy of ±0,1 mm.

11.3 Initial preparation

The conical heater shall be stabilised at the set-point temperature corresponding to the 50 kW/m^2 incident heat flux determined when setting the conical heater. The heat flux shall be (50 ± 1) kW/m^2 at (30 ± 1) mm above the exposed surface of the test specimen.

Place an aluminium foil sheet (25 µm to 40 µm in thickness) on the specimen holder, and then place the test specimen in the centre, with the exposed surface of the product facing up. The aluminium foil sheet shall be placed with the shiny side towards the test specimen. The aluminium foil sheet shall be replaced after each test. The aluminium foil sheet dimensions shall be sufficient to ensure enough support with the specimen holder during the test (recommended dimensions are: 130 mm × 130 mm).

Place the specimen holder in its location under the conical heater. The 30 mm interval between the top surface of the test specimen and the bottom surface of the conical heater shall be controlled by a gauge of the appropriate thickness and length.

11.4 Determination of exposure duration

The expansion of the product under a constant incident heat flux is related to exposure duration. The exposure duration can be defined as the time, t, to a given expansion rate threshold, or as the time to maximum expansion.

Exploratory testing shall be carried out to determine the relationship between the tested specimen and the exposure duration. For this purpose, subject specimens in several preliminary tests to different exposure durations, starting with an exposure of 2 min and 30 s and increasing the exposure duration by 30 s for each new test, until the maximum expansion is reached. The use of a product-specific exposure duration is another option.

11.5 Determination of the expansion rate

Submit each specimen to the specified irradiance (50 kW/m^2) for the defined exposure duration.

Let the test specimen cool down.

The expansion of the specimen can appear through different shapes, which can be classified into 2 groups types:

— Type 1 - Vertical expansion growth: The test sample remains flat over testing time, and expansion is globally vertical

— Type 2 - Vertical expansion growth + fall over: Measurement of expansion shall take into account the distance that was covered by the top of the sample during test.

Measure the overall final thickness (e_{of}) of the test specimen after exposure, with a ruler or a gauge. The measurement shall be determined with an accuracy of 1 mm.

Express the expansion rate of each test specimen (E_s), using Formula (1):

$$E_s = 100 \times (e_{of} - e_{im}) / e_{im} \quad (1)$$

where

E_s is the expansion rate of the test specimen, expressed in percentage (%);

e_{of} is the overall final thickness of the test specimen, expressed in millimetres (mm);

e_{im} is the average initial thickness of the test specimen, expressed in millimetres (mm).

Express the expansion rate of the material or product, as the arithmetical mean of each tested specimen, using Formula (2):

$$E_p = \frac{1}{i} \Sigma [E_s]_i \tag{2}$$

where

E_p is the expansion rate of the material or product, expressed in percentage (%);

E_s is the expansion rate of the test specimen, expressed in percentage (%);

i is the number of tested specimens.

Annex A provides guidance for the measurement of the expansion rate in case of type 1 or type 2 expansion patterns.

12 Test report

The test report shall be as comprehensive as possible and shall include any observations made during the test and shall include comments on any difficulties experienced during testing. The units for all measurements shall be clearly stated in the report.

The following essential information shall also be given in the test report:

a) name and address of test laboratory;

b) identification of the operator;

c) name and address of sponsor, if different from the manufacturer/supplier of the product;

d) name and address of the manufacturer/supplier;

e) date of the test;

f) trade name of the product and specimen identification code or number;

g) composition or generic identification;

h) details of the specimen preparation by the testing laboratory;

i) initial thickness measurements and mean initial thickness of the test specimen[1)], expressed in millimetres (mm);

j) overall final thickness of the test specimen[2)], expressed in millimetres (mm);

k) expansion rate of the test specimen[2)], expressed in percentage (%);

l) expansion rate of the material or product, expressed in percentage (%);

m) exposure duration;

n) the statement: "The test results relate only to the behaviour of the test specimens of a material under the conditions of the test; they are not intended to be the sole criterion for assessing the expansion of the material or product in use."

1) Report these items for each test specimen

Annex A
(informative)

Guidance for the determination of the overall final thickness

A.1 General

Experience has shown that PVC test specimens can expand into diverse geometrical shapes when tested according to this document. Under the conditions of the test, the charring of PVC results in a structure which is not stable, and the primary vertical expansion may deviate into other directions. This is even more relevant when the expansion is important (>1 000 %), where the collapsing of the intumescent structure is likely to occur.

This annex aims to provide guidance on how to assess the "total expansion" considering that it should reflect the effective increase of the original thickness.

A.2 Expansion geometries

The geometries of the test specimen after the test may be classified depending on the direction followed by the exposed surface during the irradiation. This expansion can occur in a vertical direction only or in a vertical direction at the beginning of the test, and then collapse or diverge in another direction.

A.3 Considerations for the measurement of e_{of}

Here are examples of overall final thickness (e_{of}) measurement of different shapes. Expansion measurement shall follow the axis of intumescence progression, eventually requesting intermediate distance measurements (h_i).