



TECHNICAL REPORT

**Characterisation, Aspects of  
Durability and Factory Production  
Control for Reactive Materials,  
Components and Products**

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# Content

## FOREWORD

- 1 Scope
  - 2 Terminology
  - 3 Characterisation
    - 3.1 Verification
    - 3.2 Identification
    - 3.3 Verification Methods and Recommendations for FPC
  - 4 Durability
    - 4.1 General Stipulations
    - 4.2 Assessment of Durability
      - 4.2.1 General
      - 4.2.2 Preparation and Conditioning of Specimens
      - 4.2.3 Testing Reactive Materials Intended for Type X Application
      - 4.2.4 Testing Reactive Materials Intended for Type Y<sub>1</sub> Application
      - 4.2.5 Testing Reactive Materials Intended for Type Y<sub>2</sub> Application
      - 4.2.6 Testing Reactive Materials Intended for Type Z<sub>1</sub> Application
      - 4.2.7 Testing Reactive Materials Intended for Type Z<sub>2</sub> Application
    - 4.3 Appropriate Durability Tests Relating to Specific Application Conditions\*
      - 4.3.1 Preparation, conditioning of specimen and evaluation of the results
      - 4.3.2 Effects of high or low temperatures
      - 4.3.3 Resistance to over-painting
        - 4.3.4 Resistance to permanent wetness
        - 4.3.5 Resistance to chemicals
        - 4.3.6 Effects in contact with metal or plastics
  - 5 Recommendation for Storage and Transport
  - Annex A1  
Examples for Test Devices and Test Procedures for Determining the Expansion Ratio
  - Annex A2  
Examples for Test Devices and Test Procedures for Determining the Expansion Pressure
  - Annex B Heat Insulating Efficiency
  - Annex C Determination of Identification Characteristics "Fingerprint"
-

## Foreword

EOTA Technical Reports are developed as supporting reference documents to European Technical Approval Guidelines and can be applicable to Common Understanding of Assessment Procedures (CUAP), EOTA Comprehension Documents or European Technical Approvals, as far as a reference is made.

EOTA Technical Reports describe some technical aspects in detail and express the common understanding of existing knowledge and experience of the EOTA bodies on a specific subject at a particular point in time.

If knowledge and experience are developing, especially during approval work, such reports can be amended and supplemented. If this happens, the effect of the changes upon the European Technical Approval Guidelines will be laid down in the relevant comprehension documents, unless the relevant European Technical Approval Guideline is revised.

This EOTA Technical Report has been prepared by a Task Group of experts from industry and test laboratories under the direction of the EOTA Working Group 11.01/04 – "Fire Stopping, Fire Sealing and Fire Protective Products". It is intended to be a source of reference for the specific parts of the ETA-Guideline 026 "Fire stopping and fire sealing products" but other product specifications may also refer to it.

## 1 Scope

This EOTA Technical Report is intended to be used as a reference source for harmonised product specifications, specifying the terminology, methods of verification and criteria for the durability of reactive materials, components and products. It is also intended for the identification of these materials, components or products and for the verification of performance within the framework of factory production control.

This Technical Report may cover issues that are not necessarily applicable for all reactive materials, components or products due to their nature and/or the way they are incorporated in construction products.

Materials, components and products exposed to specific industrial or polluted/aggressive atmosphere are not considered in this TR.

## 2 Terminology

This EOTA Technical Report uses the following specific terminology:

### 2.1 Material

A material is a substance composed of various ingredients/constituents such as sodium silicate, graphite, fibres, binders and/or other compounds which are mixed together and are then processed to create an integrated form.

### 2.2 Component (see Guidance Paper C)

A product which when combined with one or more other products makes up a kit. It may be a construction product in the sense of the CPD but this is not necessary for it to be considered as part of a kit.

### 2.3 Reactive product<sup>1</sup>

A reactive product is a product containing a reactive material which is supplied directly to works for permanent incorporation.

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<sup>1</sup> For the purpose of this document the term "reactive material" is used regardless of assessing it as a material, a component or a product.

## **2.4 Reactive material**

"Reactive material" is the generic term for materials that chemically or physically react to heat generated by a fire. The term includes both intumescent and ablative materials.

## **2.5 Intumescent material**

The term describes a material which expands, creating a foam or char, when exposed to heat in the conditions of fire to restrict the passage of heat, smoke, flames or any combination of them.

*Examples of the use of the above terminology:*

- *An intumescent MATERIAL may be composed of various ingredients/constituents such as sodium silicate, glass fibres and binders. If the material is supplied for direct incorporation into the works without further processing, it shall be regarded as a PRODUCT requiring a CE marking. Where an intumescent material requires further processing or combining with other materials before incorporation into the works, it shall not be regarded as a products but a COMPONENT.*
- *An intumescent sealant is a MATERIAL which may be integrated into construction products as a part of the finished construction product but when sold for use directly to the works, it shall be regarded as a REACTIVE PRODUCT.*

## **2.6 Ablative material:**

The term describes a material which does not expand significantly when exposed to heat but may consume energy through chemical or physical processes and only degrade slowly in the conditions of fire, remaining sufficiently stable to delay the passage of heat, smoke, flames or any combination of them.

## **2.7 Characterisation:**

The term means the identification of a reactive material, component or product and verification of its performance criteria.

## **2.8 Expansion ratio**

The expansion ratio is the quotient of the thickness of an expanded sample/specimen of an intumescent material and the original thickness of the same sample/specimen.

## **2.9 Expansion pressure**

The expansion pressure is the pressure exerted by an intumescent material as a result of intumescent activity.

## **2.10 Thermo-gravimetric analysis (TGA)**

In the thermo-gravimetric analysis the changes in the mass of a sample are measured when the sample is subjected to a given temperature-time-regime. The characteristic temperature-dependent mass changes are thus determined.

## **2.11 Differential Thermal Analysis (DTA)**

Differential thermal analysis compares the difference in heat release between the test sample and a reference sample of known characteristics when they are both subjected to an identical heating regime over a period of time.

## **2.12 Limiting Oxygen Index (LOI):**

The term describes the tendency of a material to sustain a flame. The procedure is to ignite the top of a sample and to identify the lowest oxygen concentration in a defined flowing mixture of nitrogen and oxygen which normally supports burning. The LOI value is the lowest percentage of oxygen in the mixture which sustains a flame.

## **2.13 Cone Calorimetry**

The Cone Calorimetry is a standardized test method showing the specific thermal output (heat release) of a material when subjected to a defined heating regime.

## 2.14 “Fingerprint”

The "Fingerprint" is a method or combination\* of methods (TG/TGA, DTA, IR-Spectroscopy) to identify qualitatively a specific material on the basis of its chemical composition without knowing formula and quantitative composition.

\*Note: Not all methods mentioned will be suitable at the same level and will deliver comparable results but a combination of at least two methods will.

## 2.15 Infrared spectroscopy (IR)

The Infrared spectroscopy is a fundamental method to identify structure and composition of unknown materials on the basis of specific reflection infra-red spectrum.

## 3. Characterisation

### 3.1 Verification

**Table 3-1 Suitable properties and corresponding test methods**

N°	Property	Reactive Material			
		Liquid	Paste	Solid	Powder or granules
1	Appearance (colour/ texture/gloss/ packaging)	3.1.1	3.1.1	3.1.1	3.1.1
2	Dimensions / shape	irrelevant	irrelevant	3.1.2	irrelevant
3	Viscosity	3.1.3	3.1.3	irrelevant	irrelevant
4	Density	3.1.4	3.1.4	3.1.4	3.1.4
5	Weight per unit area	irrelevant	irrelevant	3.1.5	irrelevant
6	Particle size distribution	irrelevant	irrelevant	irrelevant	3.1.6
7	Content of non-volatile components	3.1.7	3.1.7	3.1.7	3.1.7
8	Ash content	3.1.8	3.1.8	3.1.8	3.1.8
9	“Fingerprint”	3.1.9	3.1.9	3.1.9	3.1.9
10	Heat release	3.1.10	3.1.10	3.1.10	3.1.10
11	Expansion ratio <sup>2</sup>	3.1.11	3.1.11	3.1.11	3.1.11
12	Expansion pressure <sup>3</sup>	3.1.12	3.1.12	3.1.12	3.1.12
13	Flexibility <sup>4</sup>	3.1.13	3.1.13	irrelevant	irrelevant
14	LOI	3.1.14	3.1.14	3.1.14	3.1.14
15	Insulation efficiency <sup>5</sup>	3.1.15	3.1.15	3.1.15	irrelevant

<sup>2</sup> Only for intumescent products

<sup>3</sup> Only for intumescent products which generate sufficient pressure

<sup>4</sup> Only for coatings

<sup>5</sup> Only for ablative products

**NOTE 1**

*For materials which change their properties as a result of drying (e.g. liquids, pastes) it is preferable to measure the properties of the dried material but measurements on wet material under defined conditions are not precluded (e.g. density of a liquid). The thickness of dried films shall be determined according to EN ISO 2808*

**NOTE 2**

*For all selected properties not only the nominal values but also tolerances shall be declared for the product as placed on the market.*

**NOTE 3**

*If a test method described here for testing a property is not appropriate for testing the specific product and no EN test standard or ISO test standard is available, other test methods may be used but shall be described precisely in the test report of the ETA.*

**3.1.1 Appearance**

The delivered reactive material shall be examined visually. The colour, texture, gloss, surface finish, protective coatings or casings and the packaging shall be recorded clearly with appropriate terms to facilitate recognition.

**3.1.2 Dimensions and shape****3.1.2.1 Thickness**

The thickness of sheet material shall be measured on 3 specimens of approximately 10 cm x 10 cm. Strip material shall be measured on 3 specimens each approximately 50 cm in length. Measurements shall be made in 5 symmetrically arranged positions with one reading at the centre of the sheets. If the material is not available in a suitable size to meet this requirement several smaller samples may be used. The measuring equipment shall have an accuracy of 0,1 mm or 0,5 %.

If appropriate standards exist which are more suitable for the measurement of the specific material, the equipment and tolerances shall be taken from that standard e.g. EN 823 (for slabs) or EN 1849-1 or EN 1849-2 (for membranes, sheets), EN ISO 2808 (for foils and films), EN 1603;

**3.1.2.2 Other dimensions**

Other dimensions such as length and width shall be tested on at least 5 samples selected at random. Equipment and tolerances shall conform to an appropriate standard if possible, e.g. EN 12085, EN 822 or EN 1848 (for sheets and foils).

For more complex shapes individual measurement methods on critical dimensions are necessary. A description of the complex shape should be given descriptively (in writing or by a drawing). The recorded accuracy should be the same as for the thickness.

**3.1.3 Viscosity**

The test should be carried out in accordance with EN ISO 3219 or EN 12092.

**3.1.4 Density**

The preferred unit of density is kg/m<sup>3</sup>.

The test shall be carried out at standard laboratory conditions (shall be conditioned at  $(23 \pm 2)^\circ\text{C}$  and  $(50 \pm 5)\%$  rh. The volume of regular shaped samples shall be determined by calculations derived from the dimensions. The volume of irregular shaped samples shall be determined by the displacement of a suitable liquid.

The following standards are suitable:

- for solid products EN 1015-10, EN 1602, EN 323 or ISO 9427,
- for liquid products ISO 2811-1, -2, -3
- for prefabricated shapes EN 13470
- for powders and granules EN 543<sup>6</sup>

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<sup>6</sup> Some materials compact under their own weight and therefore shall be measured in accordance with an appropriate method.

### **3.1.5 Weight per unit area/length**

The unit of the weight per unit area shall be kg/m<sup>2</sup> and for the weight per unit length kg/m. The weight per unit area of sheets shall be determined by weighing at least 3 samples and measuring the linear dimensions to an accuracy of ± 1% in accordance with EN 1849-2. For small samples EN 12127 could be used. The weight per unit length shall be measured by weight of at least 3 samples and measuring the length to an accuracy of ±1%.

### **3.1.6 Particle size distribution**

The form of the particles shall be described in appropriate terms (e.g. for non-regular shaped particles such as chopped fibres, flakes etc).

The test (sieve analysis) shall be carried out in accordance with EN 1015-1. This method is suitable for regular shaped granules.

Other possible test methods are described in: EN 933-1(fine and medium sized particles); EN 12192; EN 13279-2 (fine and very fine grains); EN 1235

### **3.1.7 Content of non-volatile components**

The test shall be carried out in accordance with EN ISO 3251, if necessary EN 13820.

### **3.1.8 Ash content**

The test shall be carried out in accordance with EN ISO 3451-1.

Protective foils or coatings shall be removed before testing, if at all possible.

### **3.1.9 "Fingerprint"**

Several methods are available to determine the characteristics of reactive materials using commercially available analytical equipment (for the principles see Annex C).

These include Infra Red Analysis (IR) and Thermo-Gravimetric Analysis (TGA) or Differential Thermal Analysis (DTA). Some materials will give clear and reproducible results with one of these methods others may require a combination of these methods. In general the combination of IR and TGA or DTA delivers suitable results for clear identification. The tests shall be carried out on at least 2 specimens.

#### **NOTE 4**

*Since TGA and DTA samples are generally very small it shall be the responsibility of the test laboratory to ensure that the samples are homogeneous and representative.*

### **3.1.10 Cone-Calorimetry**

The test shall be carried out in accordance with ISO 5660-1.

The result for identification is the typical time- temperature/heat release curve.

### **3.1.11 Expansion ratio**

The expansion ratio shall be determined on at least 6 specimens to verify the ability of the material to create a foam/char in the event of fire. The mean value and the standard deviation shall be recorded in the test report.

The original thickness of the dried specimen shall be measured according to 3.1.2.1.

The thickness shall be determined after expansion. If the expansion of the material is not uniform, the thickness after expansion shall be determined from the average value of the maximum and minimum thickness.

Examples of suitable equipment and apparatus for determining the expansion ratio are shown in annex A.

Other equipment or methods are acceptable provided the results are reproducible.<sup>7</sup>

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<sup>7</sup> To achieve a reproducible result the test parameters, e.g. temperature, duration, mass of the weight on top of the specimen, if relevant, etc. shall be selected appropriately and shall be recorded.

### **3.1.12 Expansion pressure**

The expansion pressure shall be determined on at least 6 specimens to verify the ability of the material to develop pressure during foaming. The mean value and the standard deviation shall be recorded in the test report.

Examples of suitable equipment and apparatus for determining the expansion pressure are shown in annex A.

Other equipment or methods are acceptable provided the results are reproducible.<sup>7</sup>

### **3.1.13 Flexibility**

The flexibility of reactive materials shall be tested in accordance with EN ISO 1519.

### **3.1.14 Limiting Oxygen Index (LOI)**

The LOI shall be determined in accordance with ISO 4589.

### **3.1.15 Insulating efficiency**

The insulating efficiency shows the delay in heat transmission caused by the ablative material. The test shall be carried out according to annex B.

## **3.2 Identification**

The approval body or the specification writer shall select a minimum of three appropriate properties from Nos. 1 to 10, 14 or 15 of table 3-1 for the identification of the reactive material.

Where appropriate the tolerances shall be given.

## **3.3 Verification methods and recommendations for Factory Production Control (FPC)**

The manufacturer and the approval body shall agree on the required minimum number and type of tests from table 3-1 to ensure consistent production and which tests shall be carried out to verify the performance relevant for the final intended use.

At least one of the tests shall represent a relevant performance criteria (e.g. for intumescent materials expansion ratio and/or expansion pressure)

A minimum frequency of FPC testing of once per batch<sup>8</sup> for batch production and once per shift (maximum 8 hours) for continuous production process is recommended.

The FPC procedure and the tolerances for all tested criteria in the FPC shall be defined in the product specification.

## **4 Durability**

### **4.1 General Stipulations**

The durability of reactive materials may change significantly when exposed to specific conditions. This change may result in a product not achieving the expected performance. This indicates that the product probably does not meet the required resistance to the intended application conditions.

Where a product is not subject to further processing (e.g. external protection or encapsulation) to achieve its final form, it shall be tested for durability in accordance with the guidance in this document.

Where a product is subject to further processing, such as encapsulation in waterproof casings, the durability tests should be conducted in accordance with the product specification.<sup>9</sup>

The following types of final intended use conditions<sup>10</sup> are considered to be relevant for reactive materials, components and products and should be tested correspondingly:

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<sup>8</sup> Batch: The unit or quantity of production in a single complete production operation.

<sup>9</sup> This means that the product should be tested in its final form or alternatively the performance of the protective layer, foil, encapsulation etc should be separately assessed in an appropriate test.

<sup>10</sup> The designation of the use categories may change by specification writers according to their needs.



**Type X:** intended for use at conditions exposed to weathering

**Type Y<sub>1</sub>:** intended for use at temperatures below 0 °C with exposure to UV but no exposure to rain<sup>11</sup>

**Type Y<sub>2</sub>:** intended for use at temperatures below 0 °C, but with no exposure to rain nor UV<sup>11</sup>

**Type Z<sub>1</sub>:** intended for use at internal conditions with high humidity, excluding temperatures below 0 °C<sup>12</sup>

**Type Z<sub>2</sub>:** intended for use at internal conditions with humidity classes other than Z<sub>1</sub>, excluding temperatures below 0 °C.

#### **NOTE 5**

*Materials that meet requirements for type X, meet the requirements for all other types. Materials that meet requirements for type Y<sub>1</sub> also meet the requirements for type Y<sub>2</sub>, Z<sub>1</sub> and Z<sub>2</sub>. Materials that meet the requirements for type Y<sub>2</sub> also meet the requirements for type Z<sub>1</sub> and Z<sub>2</sub>. Materials that meet the requirements for type Z<sub>1</sub>, also meet the requirements for type Z<sub>2</sub>.*

## **4.2 Testing and Assessing of durability**

### **4.2.1 General**

The principle of the durability tests is to select essential physical and/or chemical properties of the product and to check whether these properties will change during exposure to defined exposure conditions.

For the ETA, the applicant and the approval body shall agree the appropriate selection of the relevant properties (relevant for the performance of the product) from table 3-1 to verify the durability.

If this Technical Report is used for preparing harmonised product standards, the specification writer should define which properties shall be considered being essential.

The test results for the unexposed and for the exposed specimens shall be compared. This stipulates that the samples for the exposure tests shall be of the same quality as for the tests of unexposed specimens. So it would be an advantage, if all specimens are taken from the same sample but also the results for unexposed specimens from approval testing may be used for this purpose.

The evaluation "no essential change in performance" shall be given if the mean value of the tested property of the exposed specimen does not deviate more than  $\pm 15\%$  of the mean value of the initial test (unexposed specimens). No single result of exposed specimens shall be less than 80% of the mean value of the initial test.

If the result falls outside these criteria, additional 4 specimens of the same sample should be exposed, tested and assessed. All four additional specimens shall fulfil the pass criteria.

### **4.2.2 Preparation and conditioning of specimens**

All samples shall be prepared as the manufacturer recommended and as intended in practice (curing and curing period, exposure with protection as intended etc). It is in the responsibility of the applicant and the Approval Body to agree the best method for testing complex shaped materials or products. The type of protection, if any, shall be recorded in the test report.

The samples shall be supported on racks or special devices (made of an inert material) to put them into the test chamber preferably in a vertical position, 20 mm separated from each other. The size of the sample shall be sufficient for at least 3 specimens for every verification test and every option model (e.g. thinnest/thickest variation of thickness) and product variation.

Before and after environmental exposure the samples and specimens shall be conditioned at a temperature of  $(23 \pm 3) \text{ °C}$  and a relative humidity (rh) of  $(50 \pm 5) \%$ .

Before and after exposure the specimen shall be weighed. The change of weight, if any, shall be recorded.

After exposure and before the post environmental testing the specimens shall be maintained in

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<sup>11</sup> Products applicable to Y<sub>1</sub> and Y<sub>2</sub> but not at temperatures below 0 °C see chapter 4.2.4.

<sup>12</sup> These uses apply for internal humidity class 5 in accordance with EN ISO 13788.

conditions of a temperature of  $(23 \pm 3)$  °C and a relative humidity of  $(50 \pm 5)$  % rh.

#### 4.2.3 Testing reactive materials intended for type X applications

Reactive materials, components and products intended for outdoor use are usually exposed to weathering – rain, UV, high temperatures in summer, frost and frost-thaw in winter. To be able to assess the fitness of the reactive material for outdoor use the following tests shall be carried out:

##### UV Chamber

The sample shall be exposed in a UV chamber to the conditions according to EN ISO 4892-3:2006, with a Type A lamp combination A.2 (table 1) according to cycle N°3 (table 4).

Air, humidified and temperature-controlled, shall be blown into the test bench. The specimens shall be sprayed with distilled or demineralised water.

A sample for at least 3 specimens as required for the verification tests<sup>13</sup> shall be stored preferably in a vertical position for 28 days in the UV chamber conducting the following procedure:

Continuous UV-irradiation for 28 days,

1 cycle takes 6 h divided as follows:

- 5 h dry phase at  $(50 \pm 3)$  °C and relative humidity below 15 % rh and
- 1 h exposed to water spray<sup>14</sup>, at  $(25 \pm 3)$  °C without controlled humidity

This cycle shall be repeated 112 times without interruption. After testing the specimens shall be visually assessed and the observations shall be recorded.

##### Environmental Chamber

After the UV chamber test the same sample shall be exposed for further two weeks to the procedure according to table 4-1 in a controlled environmental chamber without interruption<sup>15</sup>:

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<sup>13</sup> *At least 3 specimens are required for each verification test. If more than one test is intended (e.g. expansion ratio and expansion pressure) the number of specimens will grow. So the exposed sample/s should be of sufficient size.*

<sup>14</sup> Advice: Use water of approximately  $(20 \pm 5)$  °C.

<sup>15</sup> The cycle of exposure according to table 4-1 or 4-2 shall be repeated twice

**Table 4-1 Exposure condition cycle for reactive materials without temperature restriction**

period/ day	6 hour phase			
	1 <sup>st</sup> (6 hours)	2 <sup>nd</sup> (6 hours)	3 <sup>rd</sup> (6 hours)	4 <sup>th</sup> (6 hours)
1. + 2.	(20 ± 3) °C, saturated rh	(70 ± 3) °C, (20 ± 5) % rh	(20 ± 3) °C, saturated rh	(70 ± 3) °C, (20 ± 5) % rh
3. + 4.	(20 ± 3) °C, saturated rh	(30 ± 3) °C, (40 ± 5) % rh	(40 ± 3) °C, saturated rh	(30 ± 3) °C, (40 ± 5) % rh
5. + 6 + 7	(- 20 ± 3) °C	(40 ± 3) °C, saturated rh	(- 20 ± 3) °C	(40 ± 3) °C, saturated rh

**NOTE 6**

*The chamber temperature change shall be at a rate of (1.5 ± 0,5) K/min. During the period of temperature change the change of humidity is not controlled, but condensation should be avoided. The duration of temperature change is included in the duration of an exposure phase.*

After the exposure the specimens shall be cut from the sample and shall be tested according to the verification test<sup>16</sup>.

**4.2.4 Testing reactive materials intended for type Y<sub>1</sub> applications**

To be able to assess the fitness of the reactive material intended to be used for internal and sheltered external conditions (no rain) the following tests shall be carried out:

**UV Chamber**

A sample for at least 3 specimens as required for the verification tests<sup>13</sup> shall be stored preferably in a vertical position for 28 days in the UV chamber and exposed to a continuous UV-irradiation according to EN ISO 4892-3:2006, with a Type A lamp combination A.2 (table 1) at a temperature of (50 ± 3) °C and at a humidity below 15 % (dry conditions) without interruption.

After testing the sample shall be visually assessed and the observations shall be recorded.

**Environmental Chamber**

After the UV chamber test the same sample shall be exposed for two weeks to a procedure according to table 4-1 (see 4.2.3) or to table 4-2 in a controlled environmental chamber without interruption.

<sup>16</sup> Specific deviations and modifications concerning the conditioning, the shape and size of the specimen, the exposure conditions and the verification test/s shall be recorded.

**Table 4-2 Exposure condition cycle for reactive materials not for application at low temperature** <sup>17</sup>

period/ day	6 hour phase			
	1 <sup>st</sup> (6 hours)	2 <sup>nd</sup> (6 hours)	3 <sup>rd</sup> (6 hours)	4 <sup>th</sup> (6 hours)
1. + 2.	(20 ± 3) °C, saturated rh	(70 ± 3) °C, (20 ± 5)% rh	(20 ± 3) °C, saturated rh	(70 ± 3) °C, (20 ± 5)% rh
3. + 4.	(20 ± 3) °C, saturated rh	(30 ± 3) °C, (40 ± 5)% rh	(40 ± 3) °C, saturated rh	(30 ± 3) °C, (40 ± 5)% rh
5. + 6 + 7	(- 5 ± 3) °C	(40 ± 3) °C, saturated rh	(- 5 ± 3) °C	(40 ± 3) °C, saturated rh

**NOTE 6**

*The chamber temperature change shall be at a rate of (1,5 ± 0,5) K/min. During the period of temperature change the change of humidity is not controlled, but condensation should be avoided. The duration of temperature change is included in the duration of an exposure phase.*

**NOTE 7**

*If a product cannot withstand the temperature of 70 °C this may be reduced to 60 °C but for the 10 °C reduction the exposure duration of this temperature phase of the cycle shall be increased by a factor of two. This reduction is not applicable for type X.*

**NOTE 8**

*The declaration of the use categories type Y<sub>1</sub> and Y<sub>2</sub> shall include limits of temperatures, e.g. type Y<sub>1, (0/+60) °C</sub>.*

After the exposure the specimens shall be cut from the sample and shall be tested according to the verification test.

**4.2.5 Testing reactive materials intended for type Y<sub>2</sub> applications**

To be able to assess the fitness of the reactive material intended to be used for internal and sheltered external conditions (no UV, no rain) the following tests shall be carried out:

**Environmental chamber:**

A sample for at least 3 specimens as required for the verification tests<sup>13</sup> shall be stored preferably in a vertical position in a controlled environmental chamber and shall be exposed to the conditions according to table 4-1 (see 4.2.3) or to table 4-2 (see 4.2.4) for 21 days without interruption.

After the exposure the specimens shall be cut from the sample and shall be tested according to the verification test<sup>16</sup>.

**4.2.6 Testing reactive materials intended for type Z<sub>1</sub> applications**

For this test an airtight cabinet or chamber as described in EN ISO 11503 should be used.

A sample for at least 3 specimens as required for the verification tests<sup>13</sup> shall be stored preferably in a vertical position in an environmental chamber for 21 days and exposed to the following procedure without interruption:

- 8 hours at (40 ± 3) °C and saturated rh followed by
- 16 hours at (23 ± 3) °C and (50 ± 5)% rh<sup>18</sup>

<sup>17</sup> There are reactive products which are sensitive to heavy frost but are suitable for semi-exposed applications in a moderate climate – these products may be tested at the alternative temperature according to table 4-2.

After the exposure the specimens shall be cut from the sample and shall be tested according to the verification test<sup>16</sup>.

#### **4.2.7 Type Z<sub>2</sub> - testing reactive materials intended for internal (normally dry conditions) application**

For this test the specimens shall be cut out from a sample before exposure. The specimens shall be of the same dimensions and size as required for the verification tests.

At least 3 specimens as required for the verification tests<sup>13</sup> shall be stored preferably in a vertical position in an environmental chamber for 21 days and exposed to the following cycle:

- 4 h at (5 ± 3) °C and (50 ± 5)% rh
- 4 h at (23 ± 3) °C and (85 ± 5)% rh

16 h at (40 ± 3) °C and (50 ± 5)% rh This test shall be repeated for 21 cycles without interruption. After the exposure the specimens shall be tested according to the verification test.

### **4.3 Appropriate durability tests relating to specific application conditions (optional)**

The idea of these optionally additional durability assessment tests for products and kits containing reactive materials, components and products<sup>1</sup> is to make possible to assess the durability for specific application fields not covered by the use categories type X, type Y<sub>1</sub> and Y<sub>2</sub> and type Z<sub>1</sub> and Z<sub>2</sub> (see 4.1) in a uniform way.

The possibility to assess the fitness of the reactive material for special final application conditions would allow the applicant to find easily the most suitable reactive material for the specific application without further testing.

The following tests will give an approach to test and to evaluate the resistance of a reactive material to specific influences uniformly.

Reactive materials may be sensitive to specific conditions of application, especially if exposed to chemicals or permanent wetness and may influence neighbouring materials or could be influenced by neighbouring materials. The obligate basic durability assessment of a product according to 4.2 for the technical specification could be added by an assessment of durability for specific application conditions, as expected or desired. The results from the basic test should be used for the assessment

The following optional tests provide evidence for the fitness of the reactive materials concerning its performance when exposed to specific conditions of application.

#### **4.3.1 Preparation, conditioning of specimen and evaluation of the results**

The preparation of samples shall be carried out according to 4.2.2. The evaluation of the results shall follow 4.2.1.

#### **4.3.2 Effects of high or low temperatures**

##### **4.3.2.1 Effects of higher temperatures - effects of drying**

A temperature-controlled device is needed to set a constant test temperature.

The sample<sup>13</sup> shall be stored in a vertical position into the preheated device. The test sample shall be exposed to a static temperature of the expected temperature e.g. 80 °C and a relative humidity of 50 % ± 5 % for a period of 40 days.

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<sup>18</sup> In practise it is common to open the chamber door if the ambient conditions approximately meet the temperature of (23 ± 3) °C.

The test temperature, the humidity and all observations shall be recorded in the test report.

After the exposure the verification tests shall be carried out to show whether a change in performance happened caused by high temperature.

#### **4.3.2.2 Effects of low temperatures - effects of frost**

A temperature-controlled chilling device is needed to set a constant temperature at (-18 to -20) °C and at a dry atmosphere (*relative humidity of  $\leq 20$  %*).

The sample<sup>13</sup> shall be stored in a vertical position into the pre-cooled device. The test sample shall be exposed to a static temperature of -18 to -20°C and at a dry atmosphere for a period of 40 days.

The temperature, the humidity and all observations if any shall be recorded in the test report.

After the exposure the verification tests shall be carried out to show whether a change in performance happened caused by frost.

#### **4.3.3 Resistance to over-painting**

It is not possible to devise a single test for all imaginable paints. Sometimes it could be sufficient to test a representative of a family of paints e.g. epoxy resin or alcyd resin, latex or other emulsion paints etc.

Resistance to specific paints may be demonstrated using the intended paint in the following test:

For this test the specimens shall be cut out from an over-painted sample before exposure. The over-painting should be carried out according to the manufacturer's recommendation (time for drying, number of layers etc.) and as intended in practice. The specimens shall be of the same dimensions and size as required for the verification tests.

At least 3 pre-conditioned specimens (for each intended verification test) over-painted with the specified paint and allowed to dry to constant weight at a temperature of  $(23 \pm 3)$  °C and a relative humidity of  $(50 \pm 5)$ % will be tested in the verification test without removing the paint to show whether there is a change in performance happened caused by over-painting.<sup>19</sup>

#### **4.3.4 Resistance to permanent wetness**

The following short-term test is suitable to test reactive materials applied in areas where a state of permanent wetness could occur (e.g. penetration seals for liquid-lines in case of pipe rupture).

A pre-conditioned sample for at least 3 specimens (for each intended verification test) of approximately 50 mm x 50 mm, at least 20 cm<sup>2</sup>, shall be stored in desalinated water at a temperature of  $(23 \pm 2)$  °C for 4 weeks in a vertical position. After the exposure the specimens shall be stored in standard atmosphere of a temperature of  $(23 \pm 3)$  °C and a relative humidity of  $(50 \pm 5)$  % until a constant weight.

The weight of the specimens shall be recorded before and after exposure. Furthermore the test report shall record the time to come again to a constant weight and all observations if any.

After the exposure the specimens shall be cut out for the verification tests and shall be tested to show whether there is a change in performance happened caused by the water treatment.

#### **4.3.5 Resistance to chemicals**

If relevant, tests shall be carried out concerning reaction to fire of the reactive product after exposure.

##### **4.3.5.1 Liquid chemicals**

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<sup>19</sup> For intumescent materials especially the verification of the expansion ratio is of interest to avoid an obstruction of foaming caused by over-painting.

It is not possible to devise a single test for all imaginable liquid chemical, solvents and liquid cleaning agents.

If specific exposure in practice is expected, the expected liquid should be used for the exposure test. Such liquids could be: dilutions of organic or inorganic acids e.g. HCl, H<sub>2</sub>SO<sub>3</sub>, NaOH etc., of halogen-salts or alkali substances, disinfectants, liquid oxidants, degreasants, solvents, heating oil.

The following short-term test is suitable to test reactive materials applied in areas where an exposure to or a splashing with liquid chemicals, solvents and cleaning agents could occur (e.g. at the lower edge of fire-doors, penetrations or air transfer grilles in bathrooms, joint seals for rooms for disinfection in hospitals, penetrations for heating oil transporting pipes or for lines for refrigerants).

A pre-conditioned sample for at least 3 specimens (for each intended verification test) of at least 20 cm<sup>2</sup> shall be dipped completely into the specific test liquid every day for 10 seconds for a period of 3 weeks. After the third week the sample shall be stored at standard atmosphere (23 ± 3) °C and (50 ± 5) % rh for equilibrium but at least for one week.

The weight of the specimens shall be recorded before and after exposure and before testing and all observations if any.

After the exposure test the specimens shall be cut out for the verification tests and shall be tested to show whether there is a change in performance happened caused by splashing.

#### **4.3.5.2 Chemical Vapours**

It is not possible to devise a single test for all imaginable chemical vapours, sprays or aerosols.

If a specific exposure is expected in practice, the expected substance should be used for the exposure test. Such vapours could be: disinfectants, air freshener, solvents or gases for cleaning treatment

The following short-term test is suitable to test reactive materials applied in areas where chemical vapours or aerosols could occur (e.g. conservatories, dry laundries, disinfection rooms, storage rooms).

A pre-conditioned sample for at least 3 specimens (for each intended verification test) of at least 20 cm<sup>2</sup> shall be stored in an airtight chamber at a temperature of (30 ± 3) °C for 4 weeks. All 12 hours the aerosol shall be blown into the test chamber. After the exposure the specimens shall be stored in standard atmosphere of (23 ± 3) °C and (50 ± 5) % until a constant weight.

The weight of the specimens shall be recorded before and after exposure. Furthermore the test report shall contain the time to come again to a constant weight and all observations if any.

After conditioning the verification tests shall be carried out to show whether there is a change in performance happened caused by the exposure.

#### **4.3.6 Effects in contact with metal or plastics**

The basis of the following tests is a visual comparison done between exposed and unexposed specimens, in order to show the influence of the interactive effects by the change of appearance (e.g. blistering, sweating, decomposition, change of shape and colour) and to warn the observer that adverse effects could influence the function of the products.

##### **4.3.6.1 Reactive materials in permanent contact with metal**

The test shall be carried out to discern an interactive effect of reactive products in contact with metal (normal steel, aluminium, copper etc)

At least 3 unprotected metal specimens of the intended thickness and a size of approximately 20 mm x 100 mm shall be pressed directly into deep contact with a sheet of the intended reactive material of the same size. The both sheets shall be fixed on each other by small clips made of an inert material. Liquids shall be brushed or sprayed as a layer. The prepared specimens shall be stored in a horizontal position – reactive under the metal sheet - for a contact period of 40 days in the standard atmosphere of (30 ± 3) °C and (80 ± 5) % relative humidity. If other conditions are intended in practice, the intended conditions should be used.

After the test the reactive material shall be removed. The appearance of all metal specimens shall be examined for evidence of corrosion. The results shall be recorded in appropriate terms in the test report. If necessary other tests of the metal could follow (e.g. microscopy etc.)

#### **4.3.6.2.1 Reactive materials in permanent contact with plastics**

The test shall be carried out to discern an interactive effect of reactive products in contact with metal (normal steel, aluminium, copper etc)

The specimens of a size of approximately 150 mm x 40 mm made of the kind of plastics intended for the permanent contact (e.g. PVC, PE, PP, PB etc) shall be pressed directly into deep contact with a sheet of the intended reactive material of the same size. The both sheets shall be fixed on each other by small clips made of an inert material. Liquids shall be brushed or sprayed as a layer. The prepared specimens shall be stored in a horizontal position – reactive under the metal sheet - for a contact period of 40 days in the standard atmosphere of  $(30 \pm 3)^\circ\text{C}$  and  $(85 \pm 5)\%$  relative humidity. If other conditions are intended in practice, the intended conditions should be used.

After the test the reactive material shall be removed. The appearance of the plastic specimens shall be examined for changes. In addition a comparative test of tensile strength according to EN ISO 527 (exposed to contact/not exposed to contact) could be carried out to decide whether there has been a deleterious effect or not.

### **5. Recommendation for Storage and Transport**

The manufacturer of reactive materials shall give information for acceptable transport, storage conditions and suitable application conditions, on a data sheet.

The following information shall be available, where relevant:

- Temperature range
- Humidity range
- Protection against the elements and contamination
- Shelf life of the product
- Protection against mechanical damage as result of handling, storage or transport.
- Consideration of the European rules for dangerous substances

## **Annex A**

Examples for tests devices and test procedures for determining the expansion ratio and expansion pressure of intumescent materials

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### **A1 Test procedures for determining the expansion ratio**

#### **A1.1 General**

The principle of testing the expansion ratio is to expose an intumescent material to a certain temperature for a particular duration, to restrict the expansion to one direction, to measure the changed thickness after expansion (foam height) and to express this in relation to the original thickness before heat exposure.

Examples of suitable apparatus and equipment to determine the foam height are shown in figure A-1 and A-2.

Method 3 basing on the equipment of figure A-2 may be used as a combined method to determine expansion ratio and expansion pressure. But also method 1 could be modified for this purpose.



If the specific intumescent material is manufactured of more than one nominal thickness or more than one nominal density, the maximum thickness/density and the minimum thickness/density should be tested.

Pastes, mortars, stoppers and putties shall be tested preferably with a maximum thickness of approximately 5 mm and coating materials with a thickness of approximately 2 mm.

### **A1.2 Determination of the suitable test temperature**

The test temperature required for the determination of the foam height of a specific intumescent material shall be determined in pre-tests.

In order to find out the appropriate test temperature for the specific intumescent material, the temperature in the oven shall be increased in steps of 50K (300°C, 350°C, 400°C, 450°C etc). Leave the specimens in the oven until the material has completely reacted.

The duration of the test depends on the intumescent capacity of the material and can differ at different temperatures. Normally 30 minutes are considered as suitable.

It will be helpful to create an expansion-time-temperature curve. If a maximum occurs in the this curve the optimal test duration should be defined depending on the temperature to minimise scatter of results.

The determined test temperature and the relating time of exposure to heat shall be recorded and shall be used for all further tests for determining the foam height of this specific material.

### **A1.3 Test method 1 and test method 2 using the device of figure A-1**

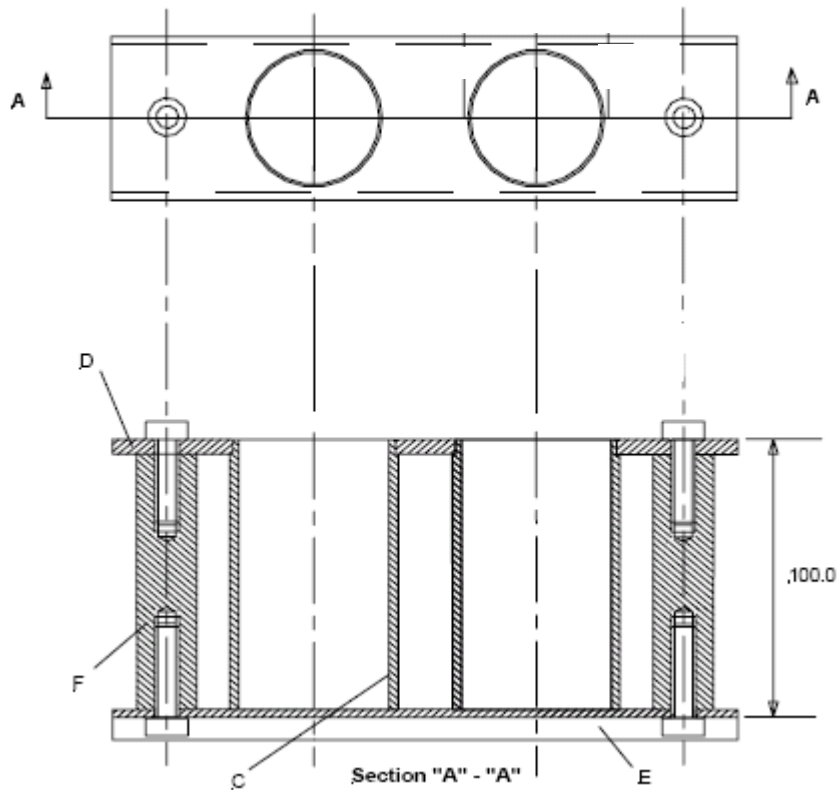
#### **A1.3.1 Preparation of the specimens**

At least six circular specimens of a diameter corresponding to the inner diameter of the specimen holder with a tolerance of  $\pm 0,5$  mm shall be cut out of the intumescent material. The thickness of the sample shall be measured with an accuracy of 0,1 mm at the centre of the disc and at four symmetrically placed positions ca. 10 mm from the edge of the specimen. The mean value should be recorded together with the standard deviation.

For intumescent materials in the form of powder or granules the weight of the specimens shall be determined.

If necessary, the thickness of the specimens may be reduced such that the foam height does not exceed the height of the specimen holder.

#### **A1.3.2 Test device**



**Figure A-1 – Test device for method 1 and method 2**

**Key**

- Pipes: according to EN 10216-5, wall thickness 2 mm
- C specimen holder
- D upper frame
- E lower frame
- F spacer bar

**A1.3.3 Test procedure**

A temperature controlled muffle oven being able to reach a stable temperature of at least 600 °C is needed. The oven shall be pre-heated to the stable temperature as required for the test<sup>20</sup>.

The specimen will be placed into the specimen holder of the test device (figure A-1).

The test device shall be placed quickly (to minimise heat loss) into the centre of the muffle oven at the particular temperature for a defined duration<sup>1</sup>. The test temperature should be re-established within 5 minutes in the oven.

Wearing protective clothing and gloves the test device shall be removed from the oven as soon as reasonably practicable after the test. The foam height will be determined within 5 minutes after the specimens have been removed from the oven.

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<sup>20</sup> as determined during the pre-test

### Test method 1:

For this method circular weights (5g; 10g, 20g, 50g, 100g etc) of a diameter corresponding to the specimen holder are put on top of the specimen before testing. After exposure to heat the height of the lower surface of the lifted weight shall be equated with the foam height.

### Test method 2:

In this tests no weights are used and the foam will expand freely in one direction in the specimen holder. After the heat exposure the measurements shall be carried out at five points symmetrically arranged with one central as done before testing (see A1.3.1).

For both methods the mean value of the measurement represents the "maximum foam height" at a certain temperature after a certain time of heat exposure. Minor voids or cavities within the structure of the foamed specimens do not have to be taken into consideration.

The expansion ratio is the quotient of the mean value of thickness of an expanded specimen of the intumescent specimen divided by the mean value of the original thickness of the same specimen before expansion.

## A1.4 Test method 3 using the device of figure A-2

### A1.4.1 Principle

The principle of method 3 is comparable to method 1 but a different geometry is used and a top die is introduced to allow uniform foaming of various geometries of specimens. The foam height is easily to read as shown in figure A-2.

### A1.4.2 Test device

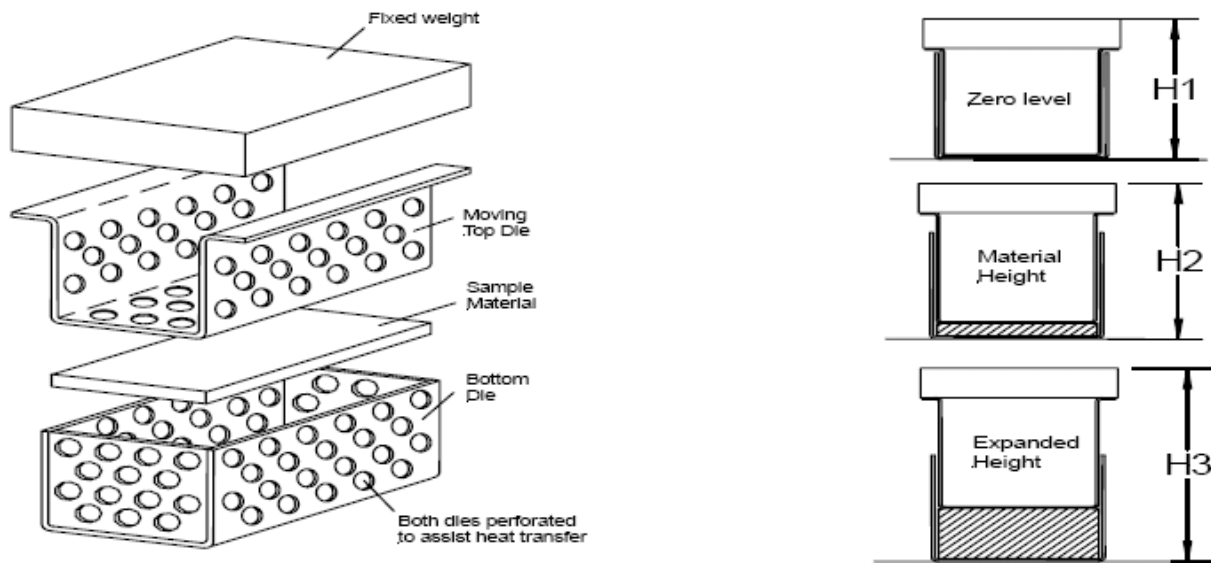
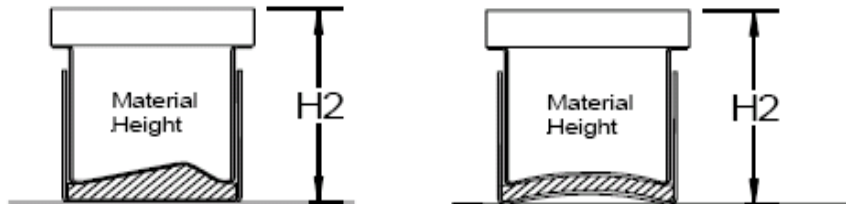


Figure A-2 –Test Apparatus method 3

### A1.4.3 Test procedure

- 1) Place the top die into the bottom die and position the fixed weight on top, then measure the heights at all four corners. Add these values together and divide by 4 to establish zero level (H1).
- 2) Prepare a sample of the material to be tested to dimensions that just fit into the bottom die. Remove top die and weight and place sample material into lower die. Replace top die and weight. Then measure the height at all four corners. Add these values together and divide by 4 to establish H2. Subtract the zero level value (H1) obtained in operation to obtain the average material height. In the case of friable materials or to prevent the intumescent material adhering to the dies, thin metallic foils of constant thickness may be placed on either face of the sample.
- 3) Place the apparatus into the centre of a small muffle furnace or oven that is set at a temperature in excess of the activation temperature of the material under test. Typically 400 deg. C.
- 4) Leave the apparatus in the furnace or oven until the material has completely activated (at least 15 minutes).
- 5) Wearing protective clothing and gloves remove the apparatus from the heating device as soon as reasonably practicable, taking care not to disturb the weight.
- 6) Measure the height at all four corners, add together and divide by 4 to obtain (H3), subtract the average zero value H1 to establish the new height of the expanded material (maximum foam height).
- 7) The expansion ratio is expressed as the quotient of maximum foam height (obtained in 6) and the original thickness of the sample (obtained in 2).



#### Note A1:

The lower faces of the top die and bottom die may be shaped to suit the contours of the material sample cross section (see examples above)

### A1.4.4 Expansion pressure

Test methods using a defined weight on top that just stops the intumescent material from moving upwards could be applied for calculating the expansion pressure via this weight and the foam height.

## A2 Tests procedures for determining the expansion pressure

### A2.1 General

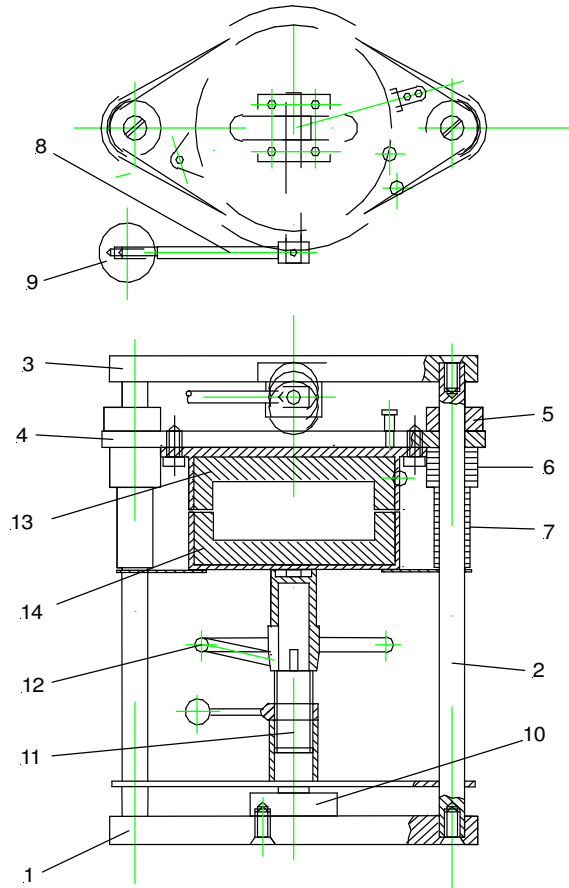
Examples of suitable apparatus and equipment to determine the expansion pressure of an intumescent material are shown in figure A-3 and A-4.

If the specific intumescent material is manufactured of more than one thickness or more than one density, the maximum thickness/density and the minimum thickness/density should be tested.

Pastes, mortars, stoppers and putties shall be tested with a maximum thickness of 5 mm, coating materials preferably of a thickness of approximately 2 mm.

## A2.2 Test method 4 and test method 5 using the device of figure A-3

### A2.2.1 Test device



**Figure A-3 - Test device for method 4 and method 5**

Key					
1	base plate	6	guide system	11	spindle
2	pillars	7	guide system	12	hand wheel
3	pressure plate	8	lever	13	heated upper pressure receptor
4	top plate	9	lever	14	heated lower pressure receptor
5	guide system	10	force transducer		

### A2.2.2 Preparation of the specimens

At least 6 circular specimens of a diameter of at least 50 mm (corresponding to diameter of the used steel ring) with a tolerance of  $\pm 0,5 \text{ mm}^{21}$ , shall be cut out the intumescent material. The thickness of the specimen shall be measured with an accuracy of  $\pm 0,1 \text{ mm}$  at the centre of the disc and at four symmetrically placed points approximately 10 mm from the edge of the specimen. The mean value shall be recorded with the standard deviation.

<sup>21</sup> The specimen should be as large as possible to reduce the edge influences, but the actual size will depend on the apparatus (e.g. ring size).

For intumescent materials being tested in the ring necessarily (e.g. pastes, powder, granules) the weight of the specimens shall be determined.

### **A2.2.3 Test procedure**

The measuring device for determining the expansion pressure, acting at given temperatures by an intumescent material, is built into a frame consisting of a base plate (1), two pillars (2) and a pressure plate (3).

The heated upper pressure receptor (13) is rigid during the test but can be swung out for cleaning purposes, is connected with the frame through a guide system (5, 6, 7). In order to enable the specimens to be quickly inserted, the top plate can be adjusted in height by 15 mm by means of the lever (8, 9).

The heated lower pressure receptor (14) transmits the force occurring during the test via a transmission device to the force transducer (10) mounted on the base plate.

The transmission device has a hand wheel adjustment that allows specimen thickness of up to 32 mm. The spindle (11) serves as a locking device for the mechanism.

### **Preparation of the test device**

In the neutral/zero position of the heating plates a pre-pressure of approximately 60 N shall exist. The starting positions for the heating plates required for the test and the appropriate spacing between them shall be established by measurement. The setting will take into account the thickness of the specimen.

The heating plates shall be spaced 1 mm greater than the height of the steel ring in use to avoid pre-pressuring the specimen and to compensate for uneven foaming at the beginning of the test .

### **Method 4:** Testing without lateral restriction

The specimens shall be placed centrally between the two aluminium foils into the apparatus according to figure A-3.

#### Note A2:

*Method 4 is not applicable for powders and granules.*

### **Method 5:** Testing with lateral restriction

The specimens shall be placed into two steel rings, which are adjusted to the size of the specimen to be tested according to item. The internal diameter of steel rings made of stainless steel should have a tolerance of + 0,2/ - 0 mm.

If possible the steel rings shall have a web height of 4 mm or 9 mm (tolerance + 0,1 mm). The specimens will then have a maximum height of 5 mm or 10 mm respectively.

The specimen shall be placed in the steel ring centrally between two aluminium foils (50µm thick) into the apparatus according to figure A-3.

#### Note A3:

*For testing specimens placed in a steel restraining ring, the heating receptors shall be spaced at a distance exceeding 1 mm from the web height of the ring.*

### A2.2.4 Measurement of the expansion pressure and evaluation of the results

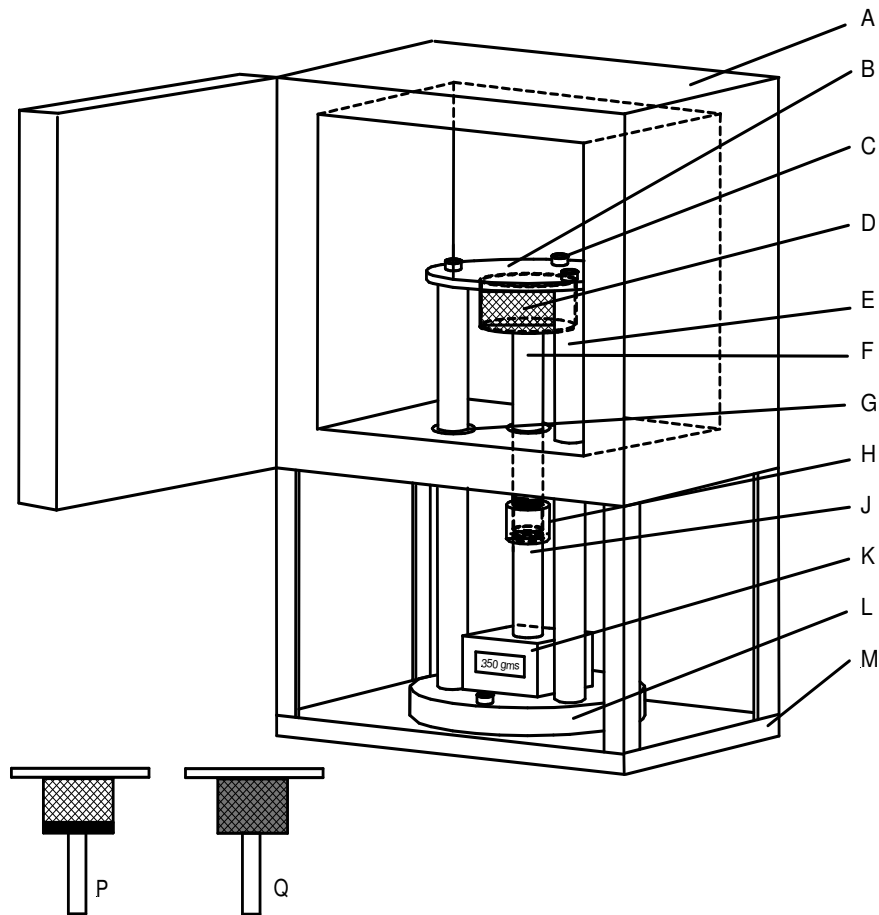
The expansion pressure [N/mm<sup>2</sup>], temperature and time elapsed shall be continuously recorded. The test is performed until the maximum pressure is clearly exceeded, but not longer than 10 min. The test report shall identify the maximum expansion pressure for each specimen thickness. The test report shall record clearly whether method 4 or method 5 was used to obtain the recorded results.

#### Note A4

*For some intumescent materials, tested according to method 5, the test results may show a significant relationship between the weight of the sample and the expansion pressure.*

### A2.3 Method 6 using the device in figure A-4

#### A2.3.1 Test device



Key					
A	Muffle furnace	F	Ceramic reaction bar	L	Base plate
B	Reaction plate	G	Ceramic fibre heat seal	M	Furnace support frame
C	Set screw	H	Threaded adjustment collar	P	Sample containing die with intumescent material ready for test
D	Sample containing die	J	Spacer bar	Q	Sample containing die with material expanded up to reaction plate.
E	Ceramic tension bars	K	Piezo-resistive force sensor		

### **A2.3.2 Test procedure method 6**

The test procedure is based on the principle of measurement of the range of expansion pressures at a fixed volume of expansion.

The test is conducted under normal laboratory conditions.

A specimen of chosen thickness is cut to a size corresponding to the internal dimensions of the specimen containing die (D)  $+0,0/ - 0,5$  mm.

A specimen containing die is selected that has a internal height that will allow the required ratio of expansion of the specimen e.g. specimen thickness 4,0 mm in a containing die of internal height of 20,0 mm will limit the expansion ratio to 5:1

The specimen is placed into the specimen containing die as shown in (P) and the reaction plate (B) is secured above the specimen containing die using set screws (C) but preloading the Piezo-resistive force sensor (K) is avoided by winding the threaded adjustment collar (H) to give a slight clearance between the reaction plate and the specimen containing die.

The Piezo-resistive force sensor is set to zero by initially finely adjusting the collar to give a small reading on the sensor and then winding back until the sensor just registers zero.

The muffle furnace door is closed and the furnace (A) is heated at a fixed rate

The time when heating commences is recorded.

The time and temperature that the specimen activates is recorded.

The time and temperature at which a positive force is registered by the sensor is recorded.

Subsequently pressures and temperatures are recorded at intervals of 2 minutes.

### **A2.3.3 Test results**

From the recordings a chart can be produced indicating pressure and temperature on the 'Y' axis versus time elapsed on the 'X' axis.

Comparing charts of identical specimen tests will provide evidence of the furnace heating curve consistency and where the heating regime is consistent the charts will show the range of pressure characteristics of a given material at a predetermined expansion ratio.

The expansion ratio may be varied by using specimen containing dies of different heights.

### **Final Note**

*If other methods will be used than described in annex A, the test device, the test procedure, the test conditions, the results and the way of interpretation shall be recorded in detail in evaluation documents of the ETA.*

*The Approval Body and the applicant shall check which method is most suitable for the specific product concerned. If a method is selected it shall be recorded in detail and shall be used without further modification. If FPC will use a different method than for approval testing, the Approval Body shall check the comparability of the results.*



## **Annex B**

### **Heat insulating efficiency**

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#### **B1 General**

This small scale furnace fire test shall be carried out at a constant temperature of 500°C (tolerance  $\pm 20$  °C).

The specimens shall be stored for drying in standard atmosphere ( $23 \pm 2$  °C and  $50\% \pm 5\%$  relative humidity) for a period of time as specified by the manufacturer.

Before exposing to heat the specimens shall be weighed. The weight shall be recorded.

#### **B2 Specimens**

The specimens shall consist of steel panels of a nominal thickness of 5 mm and a minimum of size 300 mm x 200 mm. A minimum number of two panels coated by the reactive (ablative) material shall be tested in comparison to a similar unprotected one.

#### **B3 Test Procedure**

A temperature controlled muffle oven being able to reach a stable temperature of at least 500°C is needed.

The specimen may be tested individually or together with other in one test depending on the size of the oven. The specimen shall be placed in the opening of the oven properly sealed in a vertical position such that only the ablative layer is exposed to the heat. The panel shall be mounted in a frame which forms part of one side (wall) of the furnace. The protected surface (covered with the reactive material) shall be faced to the heat.

The non-fire side shall be covered using vermiculite or calcium silicate board with a minimum of 50 mm thickness with a density of  $475 \pm 25$  kg/m<sup>3</sup>.

Two thermocouples shall be attached to the non-fire side of the steel panels. These thermocouples shall be located close to the centre in a distance of 2 cm. The thermocouples shall be of the K type according to EN 1363-1 but without a copper disc and without an insulation pad. The thermocouples shall be fixed to the back of the steel panels by welding following the ENV 13381-4: 2002.

#### **B4 Result**

The test is finished when the temperature of the two thermocouples reaches 280°C.

The time for reaching this temperature has to be recorded for the protected and the unprotected panel separately. The difference of this time is a criterion for heat transmission delay caused by the reactive material and shall be recorded.

# Annex C

## Determination of identification characteristics "Fingerprint"

### C 1 General

The "Fingerprint" is an identification method using a combination of infrared spectrum with thermal analysis of the reactive material.

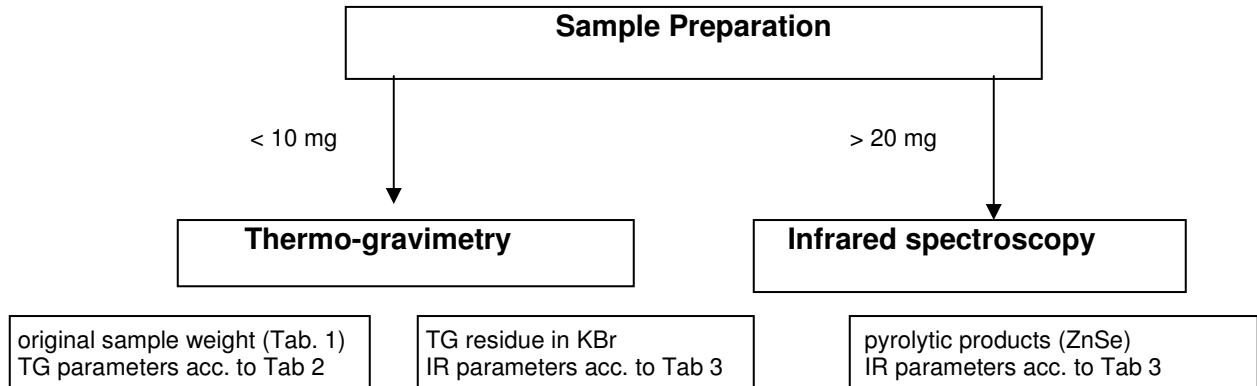


Figure C-1: Scheme of analysis

### C 2 Thermo-analytical methods (general principles see ISO 11358)

#### C 2.1 Preparation of the specimens

An identical preparation of the samples shall be provided for the thermo-analytical and infrared-spectroscopy analyses:

- Separation of a representative part quantity (ideally approx. 1g, at least approx. 30 mg) e.g. by means of a scalpel from the fire protective mass
- In the case of a highly heterogeneous sample composition: homogenizing by grinding up in a pot mill (ball mill) or in a mortar – in the case of reaction resin-bounded materials, if necessary, by using liquid nitrogen. The required quantity of original sample weights is then taken from the homogenized mass.
- Original sample weight without further treatment directly into the sample crucible according to Table C-1: analysis parameter Table C-2.

The quantity of the original weighted sample used for the TG should be chosen such that an increase in volume occurring with some materials during the process of analysis does not lead under no circumstances to an escape of components or parts of the sample from the test receptacle.

Table C-1: Recommended maximum quantity for usual sizes of test receptacle

size of receptacle / $\mu$ l	40	70	300	900
max. quantity / mg	3	4	10	30

**Table C-2: TG/TGA parameters for the analysis of fire protective materials**

Crucible	standard Alox crucible with perforated lid
Original sample weight	see Table 1
Cleansing gas / Flow	nitrogen / 50 ml/min
Range of temperature	50 – 800 °C
Rate of heating	10 K/min
Graphical representation	both TG and DTG curve

**C 3 Infrared Spectroscopy** (general principles of EN 1767)**C 3.1 Preparation of Specimen**

An identical preparation of the samples shall be provided for the thermo-analytical and infrared-spectroscopy analyses:

- Separation of a representative part quantity (ideally approx. 1g, at least approx. 30 mg) e.g. by means of a scalpel from the fire protective mass
- In the case of a highly heterogeneous sample composition: homogenizing by grinding up in a pot mill or in a mortar – in the case of reaction resin-bounded materials, if necessary, by using liquid nitrogen. The required quantity of original sample weights is then taken from the homogenized mass.
- Pyrolysis or KBr method according to instructions, analysis parameters Table C-3.

**C 3.2 Pyrolysis**

1. A typical piece of the sample material (approx. 20 – 50 mg) is placed in the lower part of a dry mini-format test tube (8 x 70 mm)
2. The tube is covered at its outer upper end with a 1 cm wide filter paper collar wetted with cold water, which is fixed by means of a test tube clamp.
3. The test tube is held with its bottom into a Bunsen flame, which is preferably carried out underneath the exhaust. The test tube remains in the flame (if necessary, turn in and out) until pyrolysis of the sample. The developing steams and vapours condense at the inner side of the test tube edge in the area of paper collar.
4. The condensate is taken with a clean glass rod and uniformly applied directly on a ZnSe crystal. The spectrum is recorded with the parameters according to Table 3 as reference against an empty crystal.

**C 3.3 KBr method**

1. 300 mg KBr powder (“spectroscopy grade”) are homogenized with the residue from the TG analysis (maximum 1 mg) e.g. in an agate mortar.
2. The powder shall be processed by pressing a KBr pellet together with the residues. The inner space of the press tool shall be evacuated for 1 or 2 minutes before pressing, in order to eliminate air and water vapour (humidity).
3. This KBr pellet will be directly compared with an plain KBr pellet in the reference sample position. **Table C-3: IR parameters for the analysis of fire protective materials**

Range of wave number	4000 – 600 cm <sup>-1</sup>
Dispersion	>4 cm <sup>-1</sup>

## Annex D

### LIST OF REFERENCE DOCUMENTS

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EN 323	Wood-based panels – Determination of density
EN 543	Adhesives- Determination of apparent density of powder and granule adhesive
EN 822	Thermal insulating products for building applications - Determination of length and width
EN 823	Thermal insulating products for building applications - Determination of thickness
EN 933-1	Test for geometrical properties of aggregates – part 1 Determination of particle size distribution – Granulometric analysis
EN 1015-1	Methods of test for mortar and masonry – Determination of particle size distribution (sieve analysis)
EN 1015-10	Methods of test for mortar and masonry – Determination of dry bulk density of hardened mortar
EN 10216-5	Seamless steel tubes for pressure purposes – technical delivery conditions – part 5: Stainless steel tubes
EN 1235	Solid fertilizer – test sieving (ISO 8397 modified)
EN 1363-1	Fire Resistance – General Requirements
EN 1602	Thermal insulating products for building applications - Determination of the apparent density
EN 1603	Thermal insulating products for building applications - Determination of dimensional stability under constant normal laboratory conditions (23 ± 3 C /50 ±5 % relative humidity)
EN 1604	Thermal insulating products for building applications - Determination of dimensional stability under specified temperature and humidity conditions
EN 1767	Products and systems for the protection and repair of concrete structures; Test methods; Infrared analysis
EN 1848-1	Flexible sheets for waterproofing – Determination of length, width and straightness
EN 1849-1,-2	Flexible sheets for waterproofing – Determination of thickness and mass per unit area
EN 12085	Thermal insulating products for building applications - Determination of linear dimensions
EN 12092	Adhesives - Determination of viscosity
EN 12127	Textiles – Determination of mass per unit area using small samples
EN 12192	Products and systems for the protection and repair of concrete structures – Granulometric analysis
EN 13279-2	Gypsum binders and gypsum plasters Part 2 Test methods

EN 13470	Thermal insulating products for building equipment and industrial installation - Determination of the apparent density of preformed pipe insulation
ENV 13381-4:2002	Contribution to Fire Resistance of structural members – Part 4: Steel elements
EN 13820	Thermal insulating materials for building applications - Determination of organic content
EN ISO 1519	Paints and varnishes – Bend test – Cylindrical mandrel
EN ISO 2808	Paints and varnishes – Determination of film thickness
EN ISO 3219	Plastics – Polymers/resins in the liquid state or as emulsion or dispersion – Determination of viscosity using a rotational viscosimeter with defined shear rate
EN ISO 3251	Paint and varnishes – Determination of non-volatile matter content
EN ISO 3451-1	Plastics – Determination of ash; method A
EN ISO 4892-1	Plastics – Method of exposure to laboratory light sources – General
EN ISO 4892-3:2006	Plastics – Method of exposure to laboratory light sources – Fluorecent UV lamps
EN ISO 13788	Hygrothermal performance of building components and building elements – Internal surface temperature to avoid critical humidity and interstitial condensation – calculation methods
ISO 554	Standard atmospheres for conditioning and/or testing - Specifications
ISO 2811	Paints and varnishes – Determination of density Part 1 Pyknometer method Part 2 Immersed body method Part 3 Oscillation method
ISO 4589	Plastics – Determination of flammability by oxygen index
ISO 5660-1	Fire tests – Reaction to fire- Cone calorimeter method
ISO 9427	Wood-based panels – Determination of density
ISO 11358:1997	Plastics -- Thermogravimetry (TG) of polymers - General principles
ISO 11503	Paints and varnishes – Determination of resistance to humidity (intermittent condensation)