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European Assessment Document for

Resilient floorings made of ethylene propylene diene (monomer) and rubber granules

This European Assessment Document (EAD) has been developed taking into account up-to-date technical and scientific knowledge at the time of issue and is published in accordance with the relevant provisions of Regulation No (EU) 305/2011 as a basis for the preparation and issuing of European Technical Assessments (ETA).

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1 SCOPE OF THE EAD

1.1 Description of the construction product

The construction products covered by this EAD are resilient floorings made of ethylene propylene diene-(monomer) (EPDM) and synthetic rubber granules (styrene-butadiene rubber (SBR)) to be used indoors comparably to EN 14041[1] (in the following referred to as "resilient floorings"). The EAD applies for granulated EPDM and SBR which can consist of pre-consumer recycled material. Polyurethane is used as binding agent, creating a binder film (coating) around granules and resulting in a hardened flooring.

The EAD does not apply for products which contain formaldehyde and pentachlorophenol or materials added during the manufacturing process or the subsequent processing which release these substances. The EAD does not apply for products containing polyvinylchloride.

The product is not fully covered by the following harmonised technical specification: EN 14041. Essential characteristics of the product regarding BWR 3 "Hygiene, health and the environment: release of dangerous substances into (indoor) air" are not fully covered in this standard.

Deviating from EN 14041, this EAD does not take into account the characteristic "Durability of reaction to fire", as in EN 14041 it is only prescribed for textile floorings with surfaces subsequently treated with a flame retardant. However, such subsequent treatment is not relevant for resilient floorings covered by this EAD.

Furthermore, some assessment methods as given in the harmonised standard are not applicable to the products as covered by this EAD.

Concerning product packaging, transport, storage, maintenance, replacement and repair it is the responsibility of the manufacturer to undertake the appropriate measures and to advise his clients on the transport, storage, maintenance, replacement and repair of the product as he considers necessary.

It is assumed that the product will be installed in accordance with the manufacturer’s instructions or (in absence of such instructions) in accordance with the usual practice of the building professionals.

Relevant manufacturer’s stipulations having influence on the performance of the product covered by this European Assessment Document shall be considered for the determination of the performance and detailed in the ETA.

1.2 Information on the intended use(s) of the construction product

1.2.1 Intended use(s)

The resilient floorings are intended to be used indoors, especially for gyms, fairs and sport facilities. They can be installed on various underlays. The resilient floorings are intended to be used only on substrates which are dry, clean and resistant to pressure. Furthermore, they should be free from cracks, oil, sealants and coatings, residual adhesive and other foreign matter.

The resilient floorings are intended to be used only inside buildings and in structures where they are protected from wetting and weathering.

The resilient floorings are intended to be installed only with a suitable adhesive. The adhesive itself is not dealt with in this EAD. If the adhesive has an influence on the performance of the product covered by this European Assessment Document regarding the reaction to fire, it shall be considered for the assessment of the performance and detailed in the ETA.

1 All undated references to standards in this EAD are to be understood as references to the dated versions listed in chapter 4.

1.2.2 Working life/Durability

The assessment methods included or referred to in this EAD have been written based on the manufacturer's request to take into account a working life of the resilient floorings for the intended use of 10 years when installed in the works. These provisions are based upon the current state of the art and the available knowledge and experience.

When assessing the product, the intended use as foreseen by the manufacturer shall be taken into account. The real working life may be, in normal use conditions, considerably longer without major degradation affecting the basic requirements for works[2].

The indications given as to the working life of the construction product cannot be interpreted as a guarantee neither given by the product manufacturer or his representative nor by EOTA when drafting this EAD nor by the Technical Assessment Body issuing an ETA based on this EAD, but are regarded only as a means for expressing the expected economically reasonable working life of the product.

2 The real working life of a product incorporated in a specific works depends on the environmental conditions to which that works is subject, as well as on the particular conditions of the design, execution, use and maintenance of that works. Therefore, it cannot be excluded that in certain cases the real working life of the product may also be shorter than referred to above.

2 ESSENTIAL CHARACTERISTICS AND RELEVANT ASSESSMENT METHODS AND CRITERIA

2.1 Essential characteristics of the product

Table 2.1.1 shows how the performance of the resilient floorings is assessed in relation to the essential characteristics.

Table 2.1.1 Essential characteristics of the product and methods and criteria for assessing the performance of the product in relation to those essential characteristics

No	Essential characteristic	Assessment method	Type of expression of product performance
Basic Works Requirement 2: Safety in case of fire			
1	Reaction to fire	2.2.1	Class
Basic Works Requirement 3: Hygiene, health and the environment			
2	Content, emission and/or release of dangerous substances	2.2.2	Description/Level
Basic Works Requirement 4: Safety and accessibility in use			
3	Gliding performance	EN 14041, clause 4.5.1	Class
4	Electrostatic behaviour	EN 14041, clause 4.6.2	Level [kV]
5	Tensile strength	2.2.3	Level [kPa]
6	Elongation at break	2.2.4	Level [%]
7	Compressive stress	2.2.5	Level [kPa]
8	Water tightness	EN 14041, clause 4.4	Description
Basic Works Requirement 6: Energy economy and heat retention			
9	Thermal conductivity	2.2.6	Level [W/(m · K)]

2.2 Methods and criteria for assessing the performance of the product in relation to essential characteristics of the product

This chapter is intended to provide instructions for TABs. Therefore, the use of wordings such as “shall be stated in the ETA” or “it has to be given in the ETA” shall be understood only as such instructions for TABs on how results of assessments shall be presented in the ETA. Such wordings do not impose any obligations for the manufacturer and the TAB shall not carry out the assessment of the performance in relation to a given essential characteristic when the manufacturer does not wish to declare this performance in the Declaration of Performance.

2.2.1 Reaction to fire

The resilient floorings shall be tested using the test method(s) relevant for the corresponding reaction to fire class in accordance with EN 13501-1. The resilient floorings shall be classified in accordance with Commission Delegated Regulation (EU) No 2016/364 in connection with EN 13501-1. The provisions given in Annex A shall be considered for mounting and fixing of the specimens when testing as well as for the extended application (EXAP) of test results.

The reaction to fire class and the conditions, for which the classification is valid, shall be given in the ETA.

2.2.2 Content, emission and/or release of dangerous substances

The performance of the product related to the emissions and/or release and, where appropriate, the content of dangerous substances will be assessed on the basis of the information provided by the manufacturer[3] after identifying the release scenarios taking into account the intended use of the product and the Member States where the manufacturer intends his product to be made available on the market.

The identified intended release scenarios for this product and intended use with respect to dangerous substances are:

IA1: Product with direct contact to indoor air.

2.2.2.1 SVOC and VOC

For the intended uses covered by the release scenario IA1 semi-volatile organic compounds (SVOC) and volatile organic compounds (VOC) shall be determined in accordance with EN 16516. The loading factor to be used for emission testing shall be 0,4 m^2/m^3.

Sampling, transport and storage of the sample proceeds essentially as described in EN ISO 16000-11. Samples shall be taken as close as possible to the time of production.

The following product parameters shall be taken into account when testing the resilient floorings:

- Each flooring with a different composition shall be tested separately.
- The highest thickness respectively area weight of the floorings shall be considered.

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3 The manufacturer may be asked to provide to the TAB the REACH related information which shall accompany the DoP with (cf. Article 6(5) of Regulation (EU) No 305/2011).

The manufacturer is **not** obliged:
- to provide the chemical constitution and composition of the product (or of constituents of the product) to the TAB, or
- to provide a written declaration to the TAB stating whether the product (or constituents of the product) contain(s) substances which are classified as dangerous in accordance with Directive 67/548/EEC and Regulation (EC) No 1272/2008 and listed in the "Indicative list on dangerous substances" of the SGDS, taking into account the installation conditions of the construction product and the release scenarios resulting from there.

Any information provided by the manufacturer regarding the chemical composition of the products may not be distributed to EOTA, to other TABs or beyond.

The testing of the specimen shall be performed with partially open edges. The length of the open edges shall be calculated in accordance with the following formula:

Open edge [m] = 0,25 [m/m^2] x area test specimen [m^2]

Once the test specimen has been produced, as described above, it shall immediately be placed in the emission test chamber. This time is considered the starting time of the emission test.

The test results shall be reported for the relevant parameters (e.g., chamber size, temperature and relative humidity, air exchange rate, loading factor, size of test specimen, conditioning, production date, arrival date, test period, test result) after 3 and/or 28 days testing.

The product performance shall be expressed in [µg/m^3 or mg/m^3] and stated in the ETA.

2.2.2.2 Polycyclic Aromatic Hydrocarbons (PAH) Content

If recycled rubber containing polycyclic aromatic hydrocarbons (PAH) from extender oils or carbon black is used, the additional assessment for the content of specific organic compounds (PAH and B[a]P) shall be performed based on the final product in accordance with the testing method described in Annex B (based on the method for the determination of PAH in polymers published by AfPS (Ausschuss für Produktsicherheit).

The product performance to be stated in the ETA shall be given as concentration of single PAH and/or as the sum of PAH in mg/kg.

2.2.2.3 *N*-Nitrosamine Content

If recycled rubber is used or nitrosamine forming agents are added to the product the additional assessment for the content of nitrosamines shall be performed based on the raw materials following the method published by DIK (Deutsches Institut für Kautschuktechnologie e.V. in Hannover, Germany, KGK, 1991):

The sample to be tested is a composite sample taking at least four incremental samples collected from different areas of a batch to represent the raw material as good as possible.

Immediately before analysis, the raw material rubber sample is cut into pieces of about 1 mm^3 particle size. About 2 g of sample are transferred to a 30 ml extraction thimble used for Soxhlet-extraction. Subsequent, extraction is performed for 24 hours at 65 °C using 75 ml *N*-nitrosamine-free methanol with 0,1 wt% ascorbic acid in a 100 ml round bottom flask containing two boiling stones made of glass.

After cooling down, 2 ml of *N*-nitrosodiisopropylamine solution (NDiPA, approximately 0,2 µg/ml) are added as internal standard. Following, the extract is evaporated with approximately 3,5 ml/min to about 5 ml using a rotary evaporator with a 40 °C water bath and 220 ± 10 mbar.

The resulting pre-concentrate is transferred to a test tube using a Pasteur pipette. The round bottom flask is rinsed twice with 1 ml *N*-nitrosamine-free methanol and the rinse solution is mixed with the preconcentrate.

By treatment with a nitrogen stream (0.05 ml/min) the solution is adjusted to 2 ml. Extracts with high oil content need chromatographic purification. The sample is analysed within 48 hours using packed columns.

The analysis of extracted *N*-nitrosamines is achieved by gas chromatography using a thermal energy analyzer (TEA) as detector. The conditions for gas chromatographic analysis are shown in table 2.2.2.3.1

Table 2.2.2.3.1 Conditions for gas chromatographic analysis of *N*-nitrosamines

Nitrosamine	NDMA, NDEA, NDPA, NDBA, NPIP, NPYR, NMOR, NDiPA	NMPA, NEPA
Column	Silanzed glas column (l = 2 m, ID = 1 mm)	
Stationary phase	10 % Carbowax 20 M, 2 % KOH on Chromosorb HAW 80/100 mesh	10 % OV 101 on Chromosorb HAW 80/100 mesh
Carrier gas	Helium	
Carrier gas flow	30 ml/min	
Sample injection	On column	
Injector temperature	200 °C	
Temperature program	125 °C 2 min isothermal 125 – 175 °C (10 °C/min) 175 °C 5 min isothermal	100 °C – 200 °C (10 °C/min)
Sample volume	5 µl	

As published in Kautschuk Gummi Kunststoffe; 44, 1991, pp. 514-21, R. Liekefeld, R. H. Schuster, G. Wünsch

The N-nitrosamines to be determined are:

- N-nitrosodibutylamine (NDBA)
- N-nitrosodiethylamine (NDEA)
- N-nitrosodimethylamine (NDMA)
- N-nitrosodipropylamine (NDPA)
- N-nitrosomethylphenylamine (NMPA)
- N-nitrosoethylphenylamine (NEPA)
- N-nitrosomorpholine (NMOR)
- N-nitrosopiperidine (NPIP)
- N-nitrosopyrrolidine (NPYR)

The content of the N-nitrosamines shall be stated in the ETA [µg/kg] on the basis of the specific level.

2.2.3 Tensile strength

The tensile strength shall be determined in accordance with the standard EN ISO 1798. A sufficient number of test specimens to obtain 5 breaks within the measuring length shall be measured. The test specimens shall have a thickness of 10-15 mm. After measurement, the tensile strength (TS) shall be calculated using equation 2.2.3.1 with F being the maximum force and A being the starting cross-section area given in square millimetres:

$$TS = \frac{F}{A} \times 10^3 \qquad (2.2.3.1)$$

The average tensile strength in kPa shall be given in ETA.

2.2.4 Elongation at break

The elongation at break shall be determined in accordance with the standard EN ISO 1798. A sufficient number of test specimens to obtain 5 breaks within the measuring length shall be measured. The test specimens shall have a thickness of 10-15 mm. After measurement, the elongation at break (E_b) stated in percent of the starting measuring length (L_0) shall be calculated using equation 2.2.4.1 with L being the measured length at break:

$$E_b = \frac{L - L_0}{L_0} \times 100 \quad (2.2.4.1)$$

The elongation at break in percent of the starting length shall be given in ETA.

2.2.5 Compressive stress

The compressive stress shall be determined in accordance with the standard EN ISO 3386-2. Three test specimens shall be tested with a width to thickness ratio of 4:1 and a side length of at least 40 mm. The compressive stress at 25 % compression in kPa shall be given in the ETA.

2.2.6 Thermal conductivity

The thermal conductivity at a reference mean temperature of 10 °C after storing the specimen in a climate of 23 °C and 50 % relative humidity until constant mass shall be determined in accordance with EN 12664.

At least 2 specimens, considering the lowest and the highest product thickness covered by the ETA, of the size of at least 200 mm x 200 mm shall be tested.

If only one product thickness is covered by the ETA, a test on one test specimen is sufficient.

If the product thickness covered by the ETA is less than the minimum test thickness in accordance with EN 12664, the specimen shall be composed of several layers.

The thermal conductivity (worst test result) shall be given in the ETA in levels with steps of 0,01 W/(m · K).

3 ASSESSMENT AND VERIFICATION OF CONSTANCY OF PERFORMANCE

3.1 System(s) of assessment and verification of constancy of performance to be applied

For the products covered by this EAD the applicable European legal act is Commission Decision 97/808/EC, as amended by Commission Decisions 1999/453/EC, 2001/596/EC and 2006/190/EC.

For all uses the applicable AVCP systems are 1, 3 or 4 depending on the conditions defined in the said Decision related to the reaction to fire classes.

3.2 Tasks of the manufacturer

The cornerstones of the actions to be undertaken by the manufacturer of the product in the procedure of assessment and verification of constancy of performance are laid down in Table 3.2.1.

Table 3.2.1 Control plan for the manufacturer; cornerstones

No	Subject/type of control	Test or control method	Minimum number of samples	Minimum frequency of control*
Factory production control (FPC) [including testing of samples taken at the factory in accordance with a prescribed test plan]				
1	Reaction to fire	2.2.1	at least 1 (depending on the test method applied)	With production start and: • EN ISO 11925-2 every three month • EN ISO 9239-1 once a year
2	SVOC and VOC	2.2.2.1	1	With production start and every year
3	PAH content	2.2.2.2	1	Every 10th batch
4	*N*-Nitrosamines content	2.2.2.3	1	Every 10th batch
5	Gliding performance	see EN 14041, clause 4.5.1	1	With production start and every 3 months
6	Electrostatic behaviour	see EN 14041, clause 4.6.2	1	With production start and every 5 years
7	Tensile strength	2.2.3	1	Once every day
8	Elongation at break	2.2.4	1	Once every day
9	Compressive stress	2.2.5	3	Once every week
10	Water tightness	See EN 14041, clause 4.4	1	With production start, after its modification and when starting a new production line
11	Thermal conductivity	2.2.6	1	With production start and every 5 years

* In case of discontinuous production these minimum frequencies shall be adapted to an equivalent frequency.

** A batch is considered to be the portioned raw material of defined particle size fraction in a maximum 3000 kg, which is then processed into the respective product.

3.3 Tasks of the notified body

The intervention of the notified body under AVCP system 1 is only necessary for products/materials for which a clearly identifiable stage in the production process results in an improvement of the reaction to fire classification (e.g., an addition of fire retardants or a limiting of organic material).

The cornerstones of the actions to be undertaken by the notified body of the product in the procedure of assessment and verification of constancy of performance are laid down in Table 3.3.1.

Table 3.3.1 Control plan for the notified body; cornerstones

No	Subject/type of control	Test or control method	Criteria, if any	Minimum frequency of control
Initial inspection of the manufacturing plant and of factory production control				
1	- Presence of suitable test equipment - Presence of trained personal - Presence of an appropriate quality assurance system and necessary stipulations taking especially account of a clearly identifiable stage in the production process results in an improvement of the reaction to fire classification	Verification of the complete FPC as described in the control plan agreed between the TAB and the manufacturer	control plan	When starting the production process, after its modification and when starting a new production line
Continuous surveillance, assessment and evaluation of factory production control				
1	- Inspection of the factory, of the production of the product and the facilities for factory production control - Evaluation of the documents concerning the factory production control including the test results - Issuing a surveillance report taking especially account of a clearly identifiable stage in the production process results in an improvement of the reaction to fire classification	Verification of the controls carried out by the manufacturer as described in the control plan agreed between the TAB and the manufacturer with reference to the process and to the product as indicated in Table 3.2.1	control plan	Once a year

4 REFERENCE DOCUMENTS

EN 12664:2001	Thermal performance of building materials and products - Determination of thermal resistance by means of guarded hot plate and heat flow meter methods - Dry and moist products with medium and low thermal resistance
EN 13238:2010	Reaction to fire tests for building products – Conditioning procedures and general rules for selection of substrates
EN 13501-1:2018	Fire classification of construction products and building elements - Part 1: Classification using test data from fire reaction to fire tests
EN 14041:2004+AC:2006	Resilient, textile, laminate and floor coverings - Essential characteristics
EN 16516:2017+A1:2020	Construction products - Assessment of release of dangerous substances - Determination of emissions into indoor air
EN ISO 1182:2020	Reaction to fire tests for products - Non-combustibility test
EN ISO 1716:2018	Reaction to fire tests for products - Determination of the gross heat of combustion (calorific value)
EN ISO 1798:2008	Flexible cellular polymeric materials - Determination of tensile strength and elongation at break
EN ISO 3386-2:1998/A1:2010	Flexible cellular polymeric materials - Determination of stress-strain characteristics in compression - Part 2: High-density materials
EN ISO 9239-1:2010	Reaction to fire tests for floorings - Part 1: Determination of the burning behaviour using a radiant heat source
EN ISO 11925-2:2020	Reaction to fire tests for building products - Part 2: Ignitability when subjected to direct impingement of flame
EN ISO 16000-11:2006	Indoor air - Part 11: Determination of the emission of volatile organic compounds from building products and furnishing - Sampling, storage of samples and preparation of test specimens
AfPS GS 2019:01	PAK – Testing and assessment of polycyclic aromatic hydrocarbons (PAH) in the course of awarding the GS mark.
KGK, 1991	Kautschuk Gummi Kunststoffe; 44, 1991, pp. 514-21, R. Liekefeld, R. H. Schuster, G. Wünsch

ANNEX A - MOUNTING AND FIXING PROVISIONS AS WELL AS EXTENDED APPLICATIONS RULES FOR THE TEST RESULTS OF THE RELEVANT REACTION TO FIRE TESTS

A.1 General

In addition to the provisions given in EN 14041 the following conditions and parameters shall be considered within testing due to the properties of the resilient floorings.

A.2 EN ISO 1182 and EN ISO 1716

These test methods are relevant for reaction to fire classes $A1_{fl}$ and/or $A2_{fl}$.
However, due to the nature of the resilient floorings the application of these both methods is not necessary, because ethylene propylene diene rubber and synthetic rubber-based products normally are not able to meet the criteria for the aforementioned classes.

A.3 EN ISO 9239-1 (Radiant panel test)

This test method is relevant for reaction to fire classes $A2_{fl}$ to D_{fl} in accordance with EN 13501-1.

A.3.1 Dimensions of the test specimens

The dimension of the test specimens shall be as prescribed in the test standard.

A.3.2 Substrate

The test specimens shall be mounted onto an appropriate standard substrate in accordance with EN 13238 representing a range of substrates in end use applications.

Other substrates (deviating from EN 13238) may also be used for testing purposes. However, in this case the test results will only be valid for the use of the resilient flooring on this specific substrate.

A.3.3 Test specimens

The following parameters shall be considered when preparing the test specimens:

- Product-variations of a product family (as defined by a certain combination of raw materials and other additives and produced in a certain production process)[4] and assembly – each product-variation and assembly (e.g., number, type and dimensions of the various layers of the resilient flooring) shall be considered within the tests,
- Colour – if there is a range of different colours but no difference in the chemical composition itself, tests with a light, a dark and a medium colour (e.g., White, Black and Red) shall be performed,
- Flame retardants – each type with the lowest amount shall be considered within the tests,
- Thickness – the highest as well as the lowest thickness of the resilient flooring shall be tested,
- Weight per unit area – the highest as well as the lowest weight per unit area shall be tested,
- Orientation – if relevant, the specimen shall be mounted and tested with lengthwise as well as with crosswise orientation,

4 To permit the TAB to apply EXAP-rules for test results within the assessment, it is recommended that the manufacturer should provide (but he is not obliged to do it) sufficient information (e.g., on the basis of the composition of the products in questions), allowing the TAB to determine which products or product variants shall be submitted to testing and to reduce the number of tests required. Otherwise, test results apply for tested configuration(s) only.

- Adhesive – either each adhesive foreseen for fixing purposes in the end-use shall be considered within the tests of the resilient flooring, taking into account the highest possible coverage of the adhesive, or the specimens shall be tested loose-laid.

The results of tests taking into consideration completely the aforementioned parameters are valid for:

- the same defined product-family and assembly as tested,
- the complete range of colours,
- any thickness between those evaluated,
- any weight per unit areas between those evaluated,
- any orientation,
- the same type of flame retardants with equal or higher amount than tested, and
- either each tested adhesive with equal or lower coverages than tested, or
- any adhesives, if the tests were carried out on loose-laid test specimens.

At least one test with any of the identified specimen configurations (based on the aforementioned parameters) shall be performed and two further tests with the most onerous specimen configuration as basis for the classification.

A.4 EN ISO 11925-2 (Small ignition source test)

This test method is relevant for reaction to fire classes B_{fl} to $E_{fl,}$ in accordance with EN 13501-1.

A.4.1 Dimensions of the test specimens and preparation

The dimension of the test specimens shall be as prescribed in the test standard.

A.4.2 Substrate

The test specimens shall be mounted onto an appropriate standard substrate in accordance with EN 13238 representing a range of substrates in end use applications.

Other substrates (deviating from EN 13238) may also be used for testing purposes. However, in this case the test results will only be valid for the use of the resilient flooring on this specific substrate.

A.4.3 Test specimens

The following parameters shall be considered when preparing the test specimens:

- Product-variations of a product family (as defined by a certain combination of raw materials and other additives and produced in a certain production process)[4] and assembly – each product-variation and assembly (e.g., number, type and dimensions of the various layers of the resilient flooring) shall be considered within the tests,
- Colour – if there is a range of different colours but no difference in the chemical composition itself, tests with a light, a dark and a medium colour (e.g., White, Black and Red) shall be performed,
- Thickness – the highest as well as the lowest thickness of the resilient flooring shall be tested,
- Weight per unit area – the highest as well as the lowest weight per unit area shall be tested,
- Orientation – if relevant, the specimen shall be mounted and tested with lengthwise as well as with crosswise orientation,
- Adhesive –each adhesive foreseen for fixing purposes in the end-use shall be considered within the tests of the resilient flooring, taking into account the highest possible coverage of the adhesive, or the specimens shall be tested loose-laid,
- Flame retardants – each type with the lowest amount shall be considered within the tests.

The results of tests taking into consideration completely the aforementioned parameters are valid for:

- the same defined product-family and assembly as tested,
- the complete range of colours,
- any thickness between those evaluated,

- any weight per unit areas between those evaluated,
- any orientation,
- each tested adhesive with equal or lower coverages than tested and
- the same type of flame retardants with equal or higher amount than tested.

The test specimens shall be tested with surface exposure. Additionally, for multilayer products with a thickness higher than 10 mm, a set of tests shall be carried out with specimens turned 90 degrees on their vertical axis with edge exposure on each different layer.

At least two tests with any of the identified specimen configurations (based on the aforementioned parameters) shall be performed and four further tests with the most onerous specimen configuration as basis for the classification.

A.5 Further advices

Since there is a wide field of possible assemblies of the resilient flooring and various parameters which may influence the reaction to fire performance, it is recommended to agree the necessary test programme between applicant, Technical Assessment Body in charge and involved test laboratory, where relevant, before commencing the reaction to fire tests.

ANNEX B - TESTING INSTRUCTIONS FOR THE DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH) IN POLYMERS

B.1 Method

B.1.1 Brief description

B.1.1.1 Standard method

A representative partial sample shall be taken of the material and cut up into pieces with a maximum size of 2-3 mm using scissors, wire cutters, etc. Then, 500 mg of the sample shall be weighed into a container and extracted with 20 ml of toluene (to which an internal standard has been added, see B.1.3.1) for 1 h at 60°C in an ultrasonic bath. An aliquot shall be taken from the extract once it has cooled down to room temperature. In the case of polymers (e.g., plastics or rubber products) for which matrix problems arise throughout the analysis, an additional purification step shall be carried out using column chromatography. Quantification shall be performed on a gas chromatograph with a mass-selective detector (GC/MSD) using the SIM method.

B.1.1.2. Method for insufficient quantities

If the total mass of material to be analysed is less than 500 mg, the procedure shall be as follows: Identical materials from the product can be combined and considered as one sample. Additional product specimens shall not be used.

If less than 50 mg of material is available for individual samples, these shall not be tested.

If the available mass of chopped-up material is between 50 mg and 500 mg, the sample shall be tested in accordance with B.1.1.1 and the quantity of toluene converted or adapted in proportion. The actual mass of the sample shall be recorded in the test report accordingly.

B.1.2 Utensils

- Ultrasonic bath with a minimum power of 200 W and a bath area of 706 cm^2, corresponding to 0,28 W/cm^2, without a basket and with an internal or external thermostat,
- Gas chromatograph with a mass-selective detector.

B.1.3 Chemicals and solutions

B.1.3.1 Chemicals

• Toluene

• Internal standards
  - Standard 1: Naphthalene-d8
  - Standard 2: Pyrene-d10 or anthracene-d10 or phenanthrene-d10
  - Standard 3: Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene

At least three internal standards shall be used; these are added to the extraction solvent (toluene).
• External standard: 18 PAH substances in accordance with the list under no. 2.3, as a mix or individually
• Petroleum ether
• Silica gel
• Sodium sulfate

B.1.3.2 Calibration solutions

The concentrations of the calibration solutions shall be chosen as follows: A three-point calibration covers a working range of 0.1 to 10 mg/kg in the samples. This corresponds to a concentration range of 2.5 to 250 ng/ml in the calibration solutions.

B.2 Sample preparation and execution

B.2.1 Extraction

500 mg of the sample shall be placed in a vial. 20 ml of toluene, previously mixed with internal standards, shall be added. The sample extraction should take place for 1 h in the ultrasonic bath at a temperature of 60 ± 2 °C. For this purpose, the vials shall be placed or suspended in the ultrasonic bath without using a basket. The vials shall then be removed, the extract shall be left to cool to 23 ± 2 °C and shaken briefly, and an aliquot shall be taken from the extract and measured either directly or following dilution with toluene.

B.2.2 Column chromatography extraction step

For some polymers (e.g., plastic or rubber products), especially those that dissolve well in toluene under the described extraction conditions, it is necessary to clean the extract using adsorption chromatography on silica gel.

For this purpose, a clean-up column with “Hahnschliff” (“stopcock”, approximately 220 mm x 15 mm) shall be filled with glass wool, 4 g of silica gel and 1 cm of sodium sulfate.

The silica gel shall be deactivated previously by adding 10% water (the corresponding volume of water shall be added to the silica gel in a glass flask, and the mixture shall be homogenised on the rotary evaporator for 1 h at standard pressure and room temperature. The silica gel shall then be stored in the sealed glass flask at room temperature).

The packed column shall be conditioned with 10 ml of petroleum ether.

The aliquot of toluene extract shall then be evaporated to a volume of approximately 1 ml on the rotary evaporator and poured into the column. The pointed flask shall be rinsed out with approximately 20 ml of eluent, which shall then also be transferred to the clean-up column. Elution shall be performed with 50 ml of petroleum ether. The collected petroleum ether eluate shall be amended with 1 ml of toluene and evaporated to a volume of approximately 1 ml under a nitrogen stream (e.g., on the TurboVap). This shall then be made up to a defined volume with toluene, and the extract shall be analysed by GC-MS.

B.2.3 Measuring procedure

The method of determination to be applied is gas chromatography with a mass-selective detector in the SIM mode.

The following 18 PAH shall be determined:
- Naphthalene
- Acenaphthylene
- Acenaphthene
- Fluorene
- Phenanthrene
- Anthracene
- Fluoranthene
- Pyrene
- Chrysene
- Benzo[a]anthracene
- Benzo[b]fluoranthene

• Benzo[k]fluoranthene
• Benzo[j]fluoranthene
• Benzo[a]pyrene
• Benzo[e]pyrene
• Indeno[1,2,3-cd]pyrene
• Dibenzo[a,h]anthracene
• Benzo[g,h,i]perylene

B.2.3.1 Measuring conditions for gas chromatography

The equipment parameters (temperatures, columns, mass traces) shall be chosen by the individual laboratory or shall be determined by the analytes.

B.2.3.2 Analysis

At least three internal standards shall be used. For these three standards, the internal standards and the correction ranges are defined as followed:

Parameter	**Internal standards with recommended reference**
• Naphthalene	Naphthalene-d8
• Acenaphthylene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Acenaphthene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Fluorene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Phenanthrene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Anthracene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Fluoranthene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Pyrene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Benzo[a]anthracene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Chrysene	Pyrene-d10 or anthracene-d10 or phenanthrene-d10
• Benzo[b]fluoranthene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Benzo[k]fluoranthene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Benzo[j]fluoranthene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Benzo[a]pyrene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Benzo[e]pyrene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Indeno[1,2,3-cd]pyrene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Dibenzo[a,h]anthracene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene
• Benzo[g,h,i]perylene	Benzo[a]pyrene-d12 or perylene-d12 or triphenylbenzene

• External calibration: for each individual PAH, at least a three-point calibration shall be carried out with reference to the internal standardisation set out above. A working range of 0.1 to 10 mg/kg is recommended.
• Concentrations above the calibration range shall be determined by diluting the extract.

Concentrations of the individual PAH shall be given in mg/kg. The sum of quantified PAH, P, shall be calculated by addition of the individual PAH concentrations, p_i, following equation:

$$\sum p_i = P \qquad (B.2.3.2.1)$$

B.2.3.3 Limit of quantification

The limit of quantification for material samples is 0.2 mg/kg per parameter.

B.2.4 Special characteristics

Naphthalene is a parameter hard to assess in products that come into contact with the skin. Experiences indicates that it is possible to identify instances of both naphthalene depletion in materials and secondary

contamination. The result obtained for naphthalene therefore only ever reflects the test specimen's current situation at the time of measurement.

B.2.5 Measuring conditions for gas chromatography

Injected volume: 1 µl pulsed splitless
Column: Rxi-PAH, 40 m, ID0.18 mm, film thickness 0,07 µm
Injector temperature: 290 °C
Transfer-line temperature: 340 °C
Initial temperature: 90 °C
Initial time: 0,7 min
Heating rate: 15 °C/min. → 120 °C
40 °C/min. → 170 °C
20 °C/min. → 340 °C