COMPLIANCE GUIDELINES

FEBRUARY 2019

COMPLIANCE SCHEME REGARDING PRODUCTS OF CONTINUOUS FILAMENT GLASS FIBRE (CFGF) USED IN GLASS FIBRE REINFORCED PLASTIC (GFRP) INTENDED TO COME INTO CONTACT WITH DRINKING WATER ACCORDING TO REQUIREMENTS OF THE GERMAN FEDERAL ENVIRONMENT AGENCY (UBA)

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CONTENT

1. ABSTRACT

Glass fibre-reinforced plastics (GFRP) are composite materials made of a polymer matrix reinforced with glass fibres. The glass fibres therein are treated with a sizing to hold individual filaments together and to promote adhesion to the polymer matrix. Glass fibre-reinforced plastics are used in drinking water contact materials. This guidance document intends to assist the supply chain actors in determining compliance of sizing formulations with the new requirements of the German Federal Environment Agency (UBA).

With the 2nd amendment to the German Drinking Water Ordinance in December 2012, the Federal Environment Agency received the task to set binding hygienic assessment principles for materials in contact with drinking water. Up to this date, the Federal Environment Agency has published guidelines and recommendations that had a less binding status. Over the next few years, the guidelines and recommendations will be translated into legally binding evaluation criteria.

According to § 17 Abs. 3 TrinkwV (German Drinking Water Ordinance), the Federal Environmental Agency can publish assessment principles and specification regarding requirements for drinking water contact materials. The Federal Environment Agency has prepared a draft of the evaluation criteria for organic materials in contact with drinking water which will replace the previous recommendations gradually.

A tiered approach has been considered for the compliance evaluation of glass fibres (GF) with sizing used to reinforce plastics intended to come into contact with drinking water. Aim of the tiered approach is to define compliance criteria the GF should fulfil to be suitable for such applications.

Tier 1. Collect compositional information for sizing down to 0.02%
(i) communication from the suppliers (e.g. declaration of compliance)
Additionally the following tests can be performed to verify availability of constituents:
(ii) screening analysis by head space for volatile substances
(iii) screening analysis by solvent extraction for semi-volatile substances

Tier 2. Perform worst case calculation with respect to relevant substances under assumption of total mass transfer for a generic glass fiber reinforced plastic (GFRP) product (e.g. 2 mm thickness, density 1.5 g/cm³, containing 50% w/w GF).

Tier 3. Perform migration modeling calculations for the GFRP with respect to relevant substances => requires knowledge about diffusion properties of the GFRP, amount of substances in the GFRP and area to volume ratio to be considered.

Tier 4. Specific migration testing at finished product level (e.g. pipes, tanks)

Worst case migration calculation is applicable for an initial migration evaluation for substances with restrictions. It is most likely suitable for substances with low initial concentration or high MTC_{tap}. These substances will not need further compliance evaluation.

>>> *After completion of tier 1 and 2 a statement of composition shall be issued by GF manufacturer or the third party performing the evaluation of recipe-specific requirements for individual substances.*

>>> *The substances with restrictions requiring further evaluation, i.e. migration modeling (tier 3) or migration testing (tier 4) shall be communicated to the expert, institute or laboratory. This will typically require setup of a non-disclosure agreement.*

About Glass Fibre Europe – EU Transparency Register n°635608817518-09.

Glass Fibre Europe, founded in 1987, is the voice of the European continuous filament glass fibre industry. It is composed of 7 companies: 3B the fibreglass company, FYSOL SAS, Johns Manville, Lanxess, Nippon Electric Glass, Owens Corning and Saint-Gobain Vetrotex. Glass Fibre Europe represents over 90% of the continuous filament glass fibre production in Europe.

2. DEFINITIONS

CFGF

Continuous Filament Glass Fibre manufactured by continuous drawing of molten glass through a "bushing" (a device fitted with calibrated holes) which forms continuous glass filaments with a defined and precisely controlled diameter. A surface treatment (sizing) is applied onto the glass filaments which are then gathered into strands.

CFGF products

The different end products are: Single End or Direct Roving, Multi-end or Assembled Roving, Chopped Strands, Textile Yarns, Technical Fabrics and Milled Fibres. With the addition of a binder, other products like Chopped Strand Mats, Continuous Filament Mats and Veils can also be produced.

Sizing

A surface treatment (usually a mixture of organic substances) applied to the CFGF composed of coupling agents, film formers and processing aids. The functions of the sizing are to allow processing of the fibres, to hold the individual filaments together and to promote adhesion of the glass fibres to the polymer matrix.

Coupling agents

Chemical substances or mixtures that have the ability to bind inorganic materials such as glass fibres to organic resins. Typical examples for coupling agents are silanes.

Film formers

Chemical substances or mixtures that have the ability to bind the glass filaments together and to protect the filament bundles against abrasion during handling, processing and storage. They usually enhance the bonding properties between fibres and polymer matrix as well. Some film formers contain a reactive polymer intended to react with the plastic matrix.

Film former processing aids

Chemical substances or mixtures that are required to obtain stable film formers.

Sizing processing aids

Chemical substances or mixtures that maintain the stability of the sizing and allow its application on the glass filaments (antifoaming agents, thickening agents, surfactants, stabilizers, emulsifiers, dispersants, pH-adjusting agents, ...)

CFGF processing aids

Chemical substances or mixtures in the sizing that allow the processing of the glass fibre strands like winding, chopping, weaving (lubricants, antistatic agents, ...).

Binder

A mixture of chemicals (usually organic substances) applied to the glass fibre strands to bind them together and form mats (Chopped Strand Mats, Continuous Filament Mats and Veils).

Compound

Engineered plastic material made from two or more constituent materials which are homogenized and are only discernible on a microscopic level.

Composite material

Engineered (plastic) material made from two or more constituent materials which remain separate and distinct on a macroscopic level within the finished structure.

Glass Fibre Reinforced Plastic (GFRP)

Compound or composite material made of plastics and CFGF products

3. INTRODUCTION

Glass fibre reinforced plastics (GFRP) are composite material made of a polymer matrix reinforced with glass fibres. The glass fibres therein are coated with a surface treatment ('sizing agent') to hold individual filaments together and to promote adherence to the polymer matrix.

3.1. Glass fibres for reinforced plastic

3.1.1. Description of CFGF Manufacturing Process

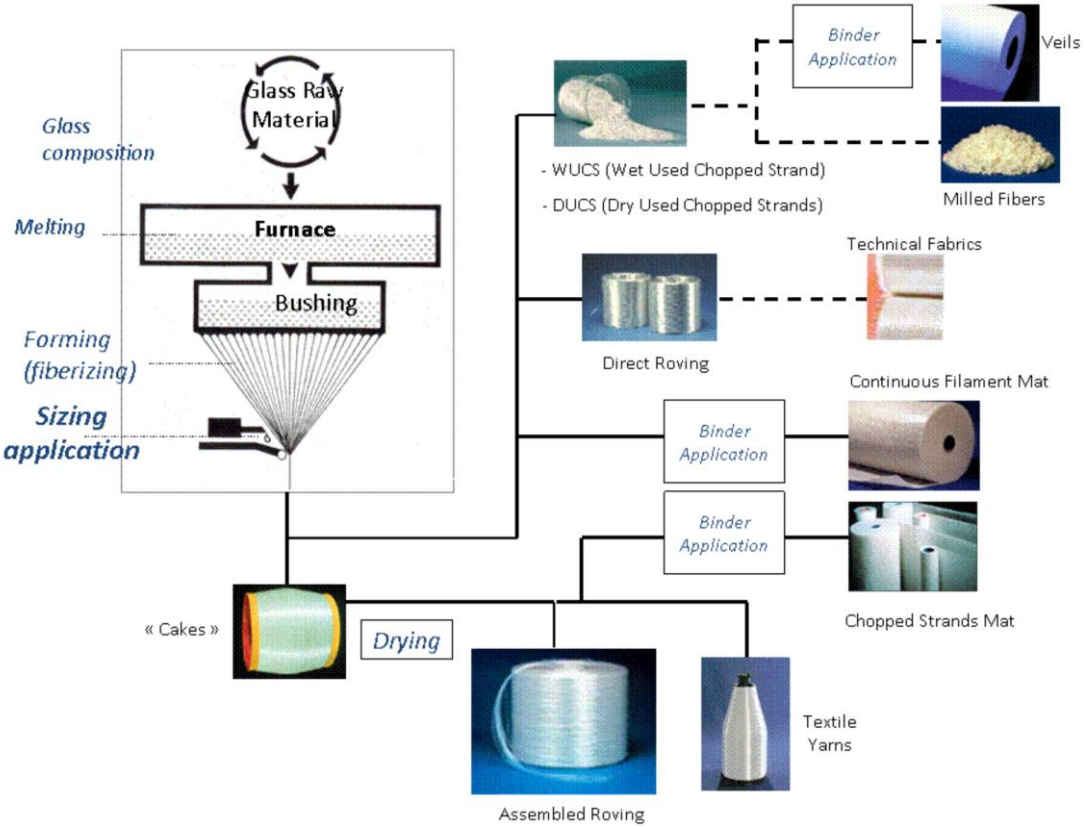

Figure 1: Schematic description of the CFGF manufacturing process

During the production of glass fibres for plastic reinforcement, molten glass flows to platinum/rhodium alloy bushings and then through individual bushing tips with orifices ranging from 0.75 to 2.0 mm. On exiting the bushing, the glass is rapidly quenched and attenuated (to prevent crystallization) into fine fibres with diameters ranging from 6 to 33 µm. As a result of the high linear speed of the glass fibres (mechanical winders or choppers pull the fibres at velocities up to 60 m/s), the very high cooling rates and the immediate interaction of the fibres with a coolant water mist, unique compositions and structures are created in the top layer of the fibre surfaces. Within milliseconds of forming and cooling, a "sizing" is applied to the glass fibre surface commonly by contacting an applicator roll carrying a layer of a mixture which may contain coupling agents, monomers or polymers and other processing aids.

During the curing process, the solvents and other volatile ingredients of the applied sizing mixtures are removed completely. Moisture content after curing is usually < 0.05%.

To some CFGF products (Chopped Strand Mat, Continuous Filament Mat, Veil), a binder is added in a second production step to bind the strands together into the desired mat or veil shape. The drying, respectively curing process, uses the following conditions:

CFGF Type	Oven Type	Curing T°	Drying + Curing Time	Type of Film Former	Final Matrix Type
Chopped Strands	Fluidized bed oven	150 - 280°C	2 - 5 min.	All	Thermoplastic, Thermoset
Direct Roving & Multi-End Roving	Hot air	110 - 140°C	24 - 36 hrs	All	Thermoplastic, Thermoset
	Radio Frequency	100 - 110°C	4 - 6 hrs	All	
Mat	Hot air	150 - 210°C	1 - 3 min.	Polyester	Thermoset

3.1.2. Sizing

Usually, sizings are aqueous chemical mixtures containing about 5% of "solids", (0.05-10%), with the rest being comprised of water. The "solids" generally consist of a number of multi-purpose components: coupling agents, film formers and processing aids. The sizing agents are composed of:

Sizing component type	Concentration (% of CFGF)
Coupling Agent	0.03 – 0.2
Film Former	0.05 – 1.0
Sizing / CFGF Processing Aids	0.001 – 0.25

Coupling Agent

The sizing generally contains an organo-functional silane commonly referred to as a coupling agent. One of the best-known properties of the silane molecules is their ability to promote adhesion between the glass surface and the different plastic matrices. They have been reported to give improvements in interfacial strength and hydrothermal resistance of the interface.

The silane coupling agents have a general chemical structure of: [R' -Si (OR)3].

The "OR" group will react with the glass surface.

The R' reactive groups of the silane may either react with the reactive groups of the film former polymer (when needed and possible) during the CFGF manufacturing process or with the reactive sites of the polymer in the curing process (thermosetting polymers) or in the high temperature extrusion and injection moulding process (thermoplastic polymers) when the sized glass fibres are introduced into the plastic matrix that they have to reinforce.

This leads to a strong network "Glass – Sizing – Plastic", in which all the partners are steadily linked through covalent and other bonds.

As to their function, coupling agents have to be considered as generating a significant effect on the properties of final reinforced plastic material.

Film Former

One of the most important components of the sizing is the film former which holds the filaments together in a strand after drying. The film former has a multifunctional role. By maintaining the strand integrity it protects the filaments from damage through fibre-fibre contact and fibre-process contact (winding, chopping etc.). Film formers are chosen to be as closely compatible to the intended polymer matrix as possible and still fulfil all the other requirements of a sizing.

The chemistry used in those film formers is generally similar to the one commonly found in adhesive applications. Film formers in their final fully reacted state are polymers.

Functions of the film former:

a) Primary function: Protection and Dispersion

Protection: Film formers bind the individual glass filaments together and protect them against abrasion during handling, storage and further processing. The primary technological function of the film formers, shaping and protecting the filament bundles, may be more or less persistent and active upon compounding into the polymer or impregnating by the polymer to create the finished plastic material.
Dispersion: The nature and the quantity of the film former on the fibres will influence the dispersion of the glass fibre filaments into the matrix during compounding or impregnating.

This is illustrated by the picture below:

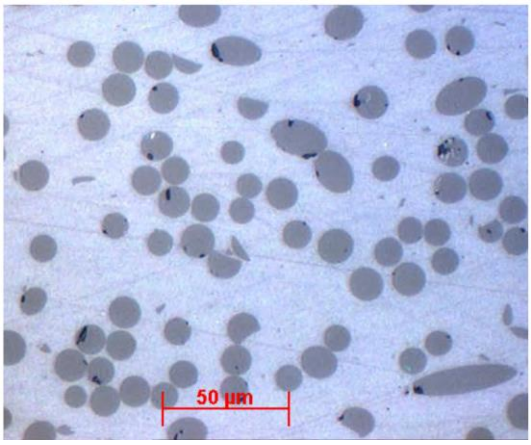

Figure 2 Polished micrograph of a typical GF reinforced plastic matrix

An optimized distribution of the fibres within the plastic matrix is essential for the manufacturing of reinforced plastics with technologically useful properties.

b) Secondary function: “Adhesive” i.e. interaction and interfacial bonding

The film former polymers are intended to remain at the interface between the glass fibre and the matrix. Many of these polymers are able to interact with the coupling agents on the glass fibre surface and/or the polymers of the plastic matrix. The type (Van der Waals, chemical bonds) of chemical link and the induced strength of adhesion have an influence on the properties of the reinforced plastic materials. The film formers are tailor–made in order to have the best compatibility with the specific type of plastics intended to be reinforced.

These two types of functions impact the properties of the reinforced plastic materials. By influencing the distribution of the fibres and/or interacting with the matrix or by chemically bonding the fibres to the matrix, the film formers may influence the mechanical properties and/or the ageing behaviour of reinforced thermoplastics. In reinforced thermosets, the ability to improve moisture content is important for the final properties.

However, it is very important to note that the interfacial bonding can only take place in combination with a suitable coupling agent. Without that, the film formers cannot sufficiently adhere to the glass fibre surface.

Examples of Film Former types:

Poly Vinyl Acetate (PVAc) and Poly Vinyl Alcohol (PVA)
Polyurethane (PUR)
Unsaturated Polyester
Epoxy resins, partly modified
Polyolefin – maleic acid modified
Poly Carboxylic acid
Poly Vinyl Pyrrolidone

<u>Sizing Processing and Curing Aids / CFGF Processing Aids</u>

a) Sizing processing and curing aids

Beyond coupling agents and film formers, other chemical substances or mixtures can be added to the sizing formulation for various purposes including, but not limited to: maintain the stability of the

sizing formulation, enhance the applicability of the sizing on the fibre, and protect the sizing from the high temperatures used in the oven.

Examples include: emulsifiers, pH adjusting agents, surfactants, antifoaming agents, antioxidants, and heat stabilizers.

b) CFGF processing aids

Beyond coupling agents and film formers, other chemical substances or preparations can be added to the sizing formulation to support mechanical processing of the fibres such as drawing and/or chopping.

Examples include: lubricants and antistatic agents.

The processing and curing aids have function only during the manufacturing process of the CFGF products and during their subsequent handling. Therefore, they may be considered production aids as defined in Regulation (EU) No. 10/2011.

Binder

The binder is applied independently from the film former and at a later production stage. It is only required for certain CFGF products like mats and veils. Its main function is to bind the glass fibre strands together. The chemistry is generally chosen to be as compatible as possible to the final composite matrix.

Examples of Binder types:
Poly Vinyl Acetate (PVAc) and Poly Vinyl Alcohol (PVA)
Polyurethane (PUR)
Unsaturated Polyester
Epoxy resins, partly modified
Polyolefin – maleic acid modified
Poly Vinyl Pyrrolidone

The binder is processed (dried and/or molten and/or cured) during a treatment step to fix the mats or veils into their intended shape.

3.2. Legal background in Germany (citation from the draft of the evaluation criteria for organic materials)

Pursuant to Section 17 subsection (2) first sentence of Drinking Water Ordinance (TrinkwV 2001) materials used for new construction or maintenance of installations for the production, treatment or distribution of drinking water and that get in contact with drinking water, must not:

1) *reduce directly or indirectly the intended protection of human health pursuant to TrinkwV 2001,*
2) *change odour or flavour of water adversely or*
3) *release substances into drinking water in quantities above what is inevitable when generally recognized rules of technology are met.*

Present evaluation criteria pursuant to Section 17 (3) of TrinkwV 2001 specify general hygienic requirements mentioned above for organic materials mentioned in the scope.

Products or components made of organic materials are assessed on the basis of the starting substances used for their production. The German Environment Agency (Umweltbundesamt (UBA)) assesses starting substances according to the principles of the European Food Safety Authority – EFSA related to food contact materials. Evaluation includes possible mass transfer and toxicological properties of the starting substance to be assessed as well as its possible contaminations and reaction and decomposition products. Assessed starting substances are mentioned in material-specific positive lists in the annexes of these evaluation criteria.

Products or components made of organic materials shall be assessed with respect to mass transfer into drinking water. For that purpose, a migration test is usually necessary by means of which starting substances including restrictions and additional requirements (possible reaction and decomposition products) shall be recorded. Besides migration water shall be evaluated concerning an impairment of odour, flavour and optics.

In addition, products or components shall be assessed with respect to promotion of microbial growth.

3.2.1. General information (chapter 5.1)

These evaluation criteria specify requirements for hygienic suitability of products or components made of organic materials in contact with drinking water. They do not contain any specifications for technical

suitability. Products or components shall be appropriate for their designated use. Relevant requirements are mentioned in the technical regulations for example.

3.2.2. Requirements for the composition (chapter 5.2)

Assessed starting substances (5.2.1)

Starting substances used for production of an organic material shall be mentioned in the valid material-specific positive list (see Annexes) according to their technological function.

In addition the substances used when manufacturing organic materials in contact with drinking water must be of a technical quality and purity that is fit for the planned and proposed purpose of the product.

Starting substances not listed (5.2.2)

Contrary to requirements in 5.2.1 the use of starting substances is possible in the following cases even if they are not mentioned on the material-specific positive list of starting substances:

a) *Low use*
   Substances with an addition below 0.02% with regard to the end product made of one substance or to the multilayer product need not be assessed and mentioned on the positive list to be used. This only applies if simultaneously the total of substances added in this way is below 0.1%.

   Note: In case of assembled products addition shall be referred to each individual component.

b) *Starting substance, its contaminations as well as possible reaction and decomposition products do not migrate into drinking water.*

Starting substances of organic materials and products in contact with drinking water do not require any toxicological assessment and thus any listing in a positive list if these substances and their contaminations including reaction and decomposition products do not pass into drinking water ("no detectable mass transfer") and are no substances of category 1A or 1B classified as carcinogenic, mutagenic or reprotoxic according to CLP Regulation No. 1272/2008 as well as substances with nanostructure.

This condition is deemed to be fulfilled if it can be shown that migration limit of MTC_{tap} = 0.1 µg/l is met for the respective product group (cf. 6.3). If the product should be intended for warm or hot water application this is also to be shown for these applications.

There are the following possibilities in order to demonstrate compliance with MTCtap= 0.1 µg/l. In all cases calculated or determined concentrations shall be converted into maximum concentrations to be expected at tap ctap (cf. 6.3.3):

- *Calculation of total transfer (100%) of substance quantity used for manufacture of considered substance passing from product into migration water (according to "Note for Guidance"):*

  $c_{calculated} = c_0 \times O/V \times L_P \times D$

  $c_{calculated}$ = *maximum possible migration of substance into migration water from product in mg/l*
  c_0 = *content of the substance in the finished product/product in mg/kg polymer*
  S/V = *surface/volume ratio of product in* dm^{-1} *according to specifications of DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04*
  L_P = *thickness of product in dm*
  D = *density of product in* g/cm^3
- *Calculation of mass transfer by means of the modelling guideline,*
- *Analytical determination of substance in migration waters of migration test as an individual material migration with appropriate analytic method. Its detection limit is at least 0.1 µg/l exclusive of analytical tolerance.*

c) *Marginal product*

   For marginal products according to chapter 5.7 no toxicological assessment of starting substances is necessary.

d) *Salts of listed acids, phenols or alcohols*

   Salts (including double salts and acid salts) of aluminium, ammonium, barium, calcium, cobalt, copper, iron, lithium, magnesium, manganese, potassium, sodium and zinc of acids, phenols or alcohols mentioned on the material-specific positive lists may be used as starting substances in addition. For the aforementioned cations the migration restriction is based on 10% of the limit values of TrinkwV 2001 and the following additional restrictions as MTCtap values:

- *Barium 7011 µg/l*
- *Cobalt 1.012 µg/l*
- *Zinc 25013 µg/l*

e) *Mixtures of substances*

Mixtures obtained by mixing listed starting substances without chemical reactions may be used.

f) *Substances > 1000 Da*

Substances having a molecular weight of more than 1,000 Da are normally not resorbed. This means that the risk to health can be classified as low. Assessment of these substances is not necessary.

g) *Prepolymers out of listed starting substances*

Prepolymers and natural or synthetic macromolecular substances and their mixtures do not require any separate listing if starting substances required for manufacture are listed. When, however, intermediate structures which have not polymerized completely and are capable of migrating into drinking water, result from polymerisation, they require an assessment and listing.

h) *colourants*

Colourants are not mentioned in the positive list as it is assumed that they do not transfer into drinking water. When colourants are used additional requirements are, however, in place (see 5.4.3).

Other components of colourant preparations shall be assessed if no other exceptional criterion is met.

i) *Ceramic fillers*

Ceramic fillers need not be mentioned in the material-specific positive lists if they correspond to evaluation criteria for enamels and ceramic materials. Evidence shall be provided on the end product (see 5.4.2).

3.2.3. Basic requirements (chapter 5.3)

General information (5.3.1)

Migration waters shall be checked for parameters such as odour, turbidity, colour and tendency to foaming. For that purpose, migration waters shall be manufactured according to specifications of DIN EN 1420: 2016-05. In this regard especially, surface/volume ratios of products are to be observed for this test.

Requirements for threshold odour number (5.3.2)

For cold, warm and hot water test the applicable threshold odour number (TON) must be met.

In addition, threshold odour numbers must indicate that there is no upward trend during testing according to DIN EN 1420: 2016-05

Requirements for turbidity and colour (5.3.3)

Parameter of turbidity is tested according to DIN EN ISO 7027: 2000-04 and parameter of colour according to DIN EN ISO 7887: 2012-04.

Requirements for tendency to foaming (5.3.4)

Tendency to foaming shall be assessed visually on migration waters according to DIN EN 1420: 2016-05.

Requirements for TOC (5.3.5)

Migration waters are prepared according to specifications of DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04. The TOC parameter is defined as a non-volatile organic carbon (NPOC) according to DIN EN 1484: 1997-08.

$c_{tap} \leq MTC_{tap,TOC} = 0.5$ *mg/l*

In addition the measured concentrations in the migration waters must indicate that there is no upward trend according to DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04.

3.2.4. Additional requirements (chapter 5.4)

Additional requirements for migration (5.4.1)

Additional requirements are defined on a material-specific basis (see Annexes).

If parameter of additional requirements to be checked is a migration restriction in form of an MTCtap value (cf. Calculation of the expected tap concentration (c_{tap})), migration shall be tested od modelled

and checked with respect to MTC_{tap} value. In this regard migration waters are manufactured according to specifications of DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04.

$c_{tap} \leq MTC_{tap}$

In addition, the measured concentrations in migration waters must not show a upward trend.

Requirements for fillers (5.4.2)

The following purity requirements shall be met for fillers mentioned in the positive lists. Soluble content of fillers in 0.07 N hydrochloric acid, determined according to DIN 53 77015 parts 1, 2, 3, 5, 6 and 13, must not be exceeded for:

lead 0.01%
arsenic 0.01%
mercury 0.0005%
cadmium 0.01%
antimony 0.005%

Purity requirements for barium sulphate: Soluble content of barium in 0.07 N hydrochloric acid, determined according to DIN 53 770 parts 1 and 4 must not be exceeded. Water-soluble content for barium sulphate, determined according to DIN ISO 787-316, must not exceed 0.4%.

Ceramic fillers not included in material-specific positive lists to be used may be assessed in accordance with evaluation criteria for enamel and ceramic fillers (chapter 6.2). Migration requirements shall be checked on the end product.

Requirements for colourants (5.4.3)

Colourants are not mentioned in material-specific positive lists. Colourants must not migrate. Further additives and adjuvants must be mentioned in the respective material-specific positive list.

The following purity requirements shall be met for the used colourants. Soluble content in 0.07 N hydrochloric acid (related to colourants) must not be exceeded for:

lead 0.01%
arsenic 0.01%
mercury 0.0005%
selenium 0.01%
barium 0.01%
chromium 0.1%
cadmium 0.01%
antimony 0.005%

Soluble content is determined in accordance with the specifications of DIN 53 770: testing of pigments, determination of parts soluble in hydrochloric acid, parts 1 to 7 as well as 13, 14 and 16.

Release of primary aromatic amines of the product in contact with drinking water must not exceed MTC_{tap} =0.1 µg/l.

Azodyes which may decompose into primary aromatic amines, classified as mutagenic, carcinogenic or reprotoxic substances of categories 1A and 1B according to CLP Regulation (EC) No. 1272/2008 must not be used.

3.2.5. Recipe-specific requirements for individual substances (chapter 5.5)

Different requirements (5.5.1)

Recipe-specific requirements for individual substances result from testing of requirements for the composition according to 5.2. Depending on starting substances recipe-specific requirement for individual substances are defined in form of

a) *migration-based requirements*
b) *maximum residual contents*
c) *specifications, purities of used starting substances*
d) *restrictions on use of starting substance or product manufactured therewith*

For some substances both a migration limit and a requirement of residual content (QM or QMA value) are indicated. In these cases only one restriction has to be tested. Preference should be given to checking MTC_{tap}.

Migration-based requirements (5.5.2)

If migration limits are defined for certain starting substances in form of MTC_{tap} values, they have to be checked. These requirements do not apply to marginal products (cf. 5.7).

For plastics in accordance with Annex A positive lists of Regulation (EU) No. 10/2011 apply. For substances preset in this Regulation with a specific migration limit (SML), the following equation applies:

MTC_{tap} = 1kg / 20l x SML

For substances with a specific migration limit (SML) in Regulation (EU) No. 10/2011 the SML-value of which, multiplied by the molecular mass ratio of the carbon molecular mass of the substance (MC) to the total molecular mass (M_{total}), is greater than or equal to 10 mg/l:

$SML \times M_C / M_{total} \geq 10$ mg/l

Migration-based requirements need not be determined. In these cases migration limit shall be covered by testing of the TOC parameter of the basic requirement.

If a SML value in the Regulation (EU) No. 10/2011 is specified as "not detectable", e.g. for acrylonitrile, migration limit is 0.1 µg/l for drinking water contact materials.

Testing of migration limit may be done by means of:

a) an analytical migration test according to 6.3

b) modelling of mass transfer by means of the modelling guideline [1] (cf. 6.3.2)

Determined concentration will be converted according to 6.3.3 into concentration to be expected at the tap (c_{tap}).

$c_{tap} \leq MTC_{tap}$

In addition the measured concentrations must not show an upward trend.

Maximum residual content (5.5.3)

For substances with a QM or QMA limit a review of the residual content of the substance in the product is required. The QM and QMA limits apply independently of the product group of the organic material.

Other requirements (5.5.4)

Beside requirements for migration or their residual content also requirements of specifications or purities in regard to starting substance or restrictions on use for the starting substance in a product may be laid down (cf. 4.2).

3.2.6. Requirements for the testing of propagation of microorganisms (chapter 5.6)

Testing of the products with respect to promotion of microbial growth is done according to DIN EN 16421: 2015-05. For using the three procedures described in the standard the following restrictions apply.

Procedure 3 (MDOD method) shows a too high detection limit compared to other procedures. The procedure is not suitable to assess products intended to be used with disinfectant-free

drinking water. In Germany many drinking waters are distributed without the addition of chlorine or other disinfectants. For this reason, testing according to another of the two procedures (BPP procedure or volumetric procedure) is necessary for the use in Germany.

The BPP procedure (procedure 1) is not suitable for the testing of multi-layer composite products (e.g. pipes or hoses) as also surfaces not being in contact with drinking water normally will get in touch with migration water.

Multi-layer composite products (e.g. pipes or hoses) shall be tested with procedure 2 in the test module for pipes and hoses.

3.2.7. Marginal products (chapter 5.7)

Products for which a conversion factor smaller than or equal to 0.01 d/dm shall apply (cf. Table 6), shall be considered as marginal products.

The starting substances of these products need not be assessed or mentioned in a positive list.

Parameters of basic requirements (cf. 5.3) shall be observed.

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1 Guideline relating to mathematical estimate of migration of individual substances from organic materials into drinking water

Requirements for the migration of individual substances as well as additional requirements do not apply to these products and a corresponding test is therefore not necessary.

Testing the propagation of microorganisms according to 6.4 is, however, necessary.

3.2.8. Multilayer products (chapter 5.8)

Multilayer products may be composed of different layers. These layers are firmly attached with each other.

Assembled products, however, are separated into their components and assessed on a material-specific basis for their suitability relating to drinking water hygiene.

The individual layers of a multilayer product shall be assessed on a material-specific basis in accordance with the annexes of these evaluation criteria. Migration limits of all layers shall be assessed. An exception are multilayer products with a total barrier 19, in this case only the layers on the side facing drinking water shall be assessed.

It may be that layers of a product consist of different materials. Composition of the individual layers must correspond to the respective material-specific positive list.

To check migration limits of layer not directly in contact with drinking water there are the following possibilities:

- Consideration of total mass transfer (100% substance migration) or

- Mathematical estimate of migration into drinking water of extended warm water test after a storage time of 30 days at room temperature or

- Performance of warm water test with 22 migration periods irrespective of intended application after a storage time of 30 days at room temperature or

- Separate test of the individual layers.
For that purpose it is to be ensured that addition of migration results of the individual layers corresponds to multilayer product. Same migrants of all layers to be assessed are to be added for the evaluation of MTCtap.

3.3. Four Member States Initiative (4MS)

Four EU Member States France, Germany, the Netherlands and the United Kingdom (4MS) announced in January 2011 that they have formalised arrangements to work together on this important aspect of the regulatory frameworks they have in place to ensure the hygienic safety of drinking water. The so called 4MS Initiative aims harmonizing the compliance evaluation procedure for drinking water contact materials at EU level.

The UBA accepts substance assessments of other EU Member States if they have been performed in accordance with specifications of the document of the 4MS cooperation for organic substances. These substances are also included in the relevant positive lists.

An essential element of the regulatory arrangements for control of the hygienic performance of organic Products in contact with Drinking Water (PDW's) is the examination and approval of the substances used for the production of these products. The goal of the 4MS Initiative is to have a Positive List of substances that are permitted for the production of organic materials, which is accepted by all MS's. This is in addition to the substances authorized for use in food contact materials (FCM) according to Regulation (EU) No. 10/2011, as these are included as permissible for use in PDW's.

Candidate substances for this Positive List (the "Core List") are the substances on the existing national Positive Lists in use in France, Germany and the Netherlands, and which had been collated earlier in the "Combined List". The substances on the Combined List can only be transferred to the Core List after they have been reviewed, therefore the Combined List is the list of substances under review.

3.3.1. Core List (list of confirmed substances)

The 4MS Initiative has agreed procedures for the evaluation of organic substances in use in products in its report "Common Approach on Positive Lists for Organic Materials", published in December 2011, and revised in March 2016. The Common Approach sets out the process to be followed for the assessment and acceptance of substances. Substances approved by this procedure are included in the "Core List", which is the 4MS-Positive List of substances that can be used for the production of PDW's.

Substances from the Combined List which have been assessed according to the criteria for approval by one of the MS's, and for which the positive opinion was accepted by the other MS's, have been

transferred now onto the Core List. The Core List therefore only contains confirmed substances. In addition, substances from the Combined List for which a favourable EFSA opinion was already available, or for which other adequate information was available, have also been transferred onto the Core List.

3.3.2. Combined List (list of substances under review)

The first version of the Combined List was published in 2011. This was a compilation of substances that appeared on the national lists and which did not appear in the Union List of Regulation (EU) No. 10/2011 (referred to as: “FCM-List of 10/2011”). Pigments and colourants currently used in the MS’s are not included in the Combined List. The Combined List is now updated and a revised version is now published

3.3.3. Obsolete List (list of obsolete substances)

Some substances from the Combined List that are not known to be used anymore in certified or approved PDW’s, have been transferred onto the ‘Obsolete List’. This List is found in Annex C. In situations where it appears that a substance has inadvertently been miss-classified as being ‘obsolete’, then this substance can be returned to the Combined List. Substances that are on the Obsolete List will eventually be deleted.

3.3.4. Actions required by Industry

Should Industry notice substances on the Obsolete List, which they still use in certified or approved products, Industry can inform the relevant MS, making reference to the certificate or approval. The substance will be returned to the Combined List.

For the substances on the Combined-List, Industry is asked to submit dossiers to provide data in accordance with the requirements as stated in the 4MS Common Approach on Positive List for Organic Materials, including relevant migration data into drinking water, to one of the 4MS’s. The 4MS will provide the deadline dates for submission of dossiers for material types shortly.

4. COMPLIANCE EVALUATION PROCEDURE (TESTING OR MODELLING)

4.1. Formulation-dependent requirements / Recipe review

Formulation-dependent requirements are related to the composition of the final drinking water contact material, which requires tracing back compositional information throughout the value chain. This process is laborious and requires cooperation of all affected parties.

Collection of full compositional details for a glass fibre with sizing typically requires support of an independent third party due to confidentiality constrains.

The following information is necessary for recipe review:

- description of exact structure of product/component
- designation of material type/material types,
- listing of all starting substances for manufacture of product (monomers, additives, polymer production aids and other starting substances) including compilation of chemical designations, commercial names, CAS no., technological functions, required quantities, suppliers and safety data sheets

It has to be determined by means of recipe review if requirements for the composition (5.2) are met. For that purpose, it has also to be checked if restrictions on use, e.g. with respect to technological function and specifications of listed substances, are met.

For recipe review of multilayer products recipe review is done separately for each layer.

For multilayer products with barrier specifications in chapter (5.8) apply.

As soon as all substances which require safety assessment are known, allocation to the following categories is required.

1) intentionally added substances with a MTC_{tap} listed in the mandatory evaluation criteria, Annex I of Regulation (EU) No 10/2011 or the 4MS core list and combined list.
2) intentionally or non-intentionally added substances which are not listed, i.e. the non-detectable migration (DL = 0.1 µg/l) applies.

4.2. Migration evaluation

To complement the compliance assessment most likely it will be necessary to perform a migration evaluation of the final product (glass fibre-reinforced plastics). The purpose of such migration evaluation would be to verify compositional and migration limitations of substances listed or not in the mandatory evaluation criteria respectively in referenced regulations like Annex I of Regulation (EU) No 10/2011 including known non-intentionally added substances (NIAS) such as reaction products, oligomers and impurities.

4.2.1. Calculation of total mass transfer

Calculation of total transfer (100%) of substance quantity used for manufacture of considered substance passing from product into migration water (according to “Note for Guidance”):

$$c_{calculated} = c_0 \times O/V \times L_P \times D$$

$c_{calculated}$ =	maximum possible migration of substance into migration water from product in mg/l
c_0 =	content of the substance in the finished product/product in mg/kg polymer
S/V =	surface/volume ratio of product in dm^{-1} according to specifications of DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04
L_P =	thickness of product in dm
D =	density of product in g/cm^3

4.2.2. Migration Modelling

Instead of an experimental test migration for recipe-specific requirement for individual substances can also be estimated using the Modelling Guideline [2]. This requires that material or product-specific parameters for modelling are mentioned in the Modelling Guideline.

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2 https://www.umweltbundesamt.de/en/document/modelling-guideline
Guideline for the Mathematical Estimate of the Migration of Individual Substances from Organic Material in Drinking Water (Modelling Guideline); 7. October 2008

In order to perform migration modelling the initial concentration respectively residual amount of migrating substances in the glass fibre with sizing must be determined by total extraction or known due to communication with suppliers provided the quantity of the substance does not change during the manufacture and processing of the product.

Compositional data may be obtained by non-target screening analysis of the glass fibres with sizing.

The migration modelling guideline is under review.

Modelling must consider the respective test conditions (test temperature and test cycle). The concentration profile for the previous test period is used to calculate the migration for the following test period. This is described in detail in the modelling guideline.

If result of modelling does not correspond to the specific requirement (MTC_{tap}) compliance may still be demonstrated by experimental testing. The results of experimental tests must be weighted higher than those of the modelling.

4.2.3. Migration Testing

Migration test is done according to standards of DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04 and DIN EN 1420: 2016-05. In accordance with scope of product migration test shall be carried out as a cold water test at 23°C ± 2°C (all products) and possibly as a warm water test 60°C ± 2°C (products of drinking water installation) or hot water test 85°C ± 2°C (special hot water applications). Surface/volume ratios to be adjusted for the test according to standards.

The migration waters provided for analysis have to be tested with respect to parameters resulting from basic requirements, additional requirements and recipe-specific requirements for individual substances as well as from starting substances not listed for intended product group.

Validated analytic procedures should normally be followed in testing the migration waters. Where no suitable analytic method currently exists for a particular substance an analytic method of suitable accuracy which enables an assessment of the recorded concentration to be made, may be applied. If there is no analytic method available for individual substances an estimate of the migration for this substance has to be performed, e.g. calculation of total transfer or modelling.

4.2.4. Calculation of expected concentration at the tap (c_{tap})

The expected tap concentrations (c_{tap}) differ for the various product groups according to conversion factors F_C.

$$c_{tap} = F_C \times c_{measured\ or\ modelled} / (S/V \times t)$$

F_C:	Conversion factor according to Table 6
$c_{measured\ or\ modeled}$:	In the migration water according to DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04 measured or estimated concentration according to 6.3.2
S/V:	Surface/volume ratio according to DIN EN 12873-1 2014-09 or DIN EN 12873-2: 2005-04 in accordance with test run
t:	Duration of the migration period according to DIN EN 12873-1: 2014-09 or DIN EN 12873-2: 2005-04

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