Notes on Applications for the Inclusion of Substances in BfR Recommendations XXXVI, XXXVI/1, XXXVI/2 or XXXVI/3 (“Paper Recommendations”)

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The below-listed notes do not constitute finalised test criteria. Each submitted application is tested individually in line with the current level of knowledge, which means that supplementary requirements can result.

1 Introduction

When applying for the inclusion of substances in the BfR recommendations on materials intended for contact with foodstuffs, a dossier must be presented which permits reliable conclusions on the type and quantity of the transfer of application-relevant substances from the paper into the foodstuffs that come in contact with it under worst case application conditions and which also contains the applicable toxicological data records. This data is determined on the basis of the questionnaire of the European Food Safety Authority (Note for Guidance), which was developed for substances intended for use in the manufacture of plastics intended to come into contact with foods. As different information is required in some points when making an application for substances for the manufacture of papers, cardboards and paperboards, the BfR has compiled some supplementary notes on this.

When evaluating the chemicals for which an application is submitted, not only the active substances themselves are taken into account but also their impurities, reaction and degradation products. They are included in the term “substances” used below.

Gravimetric testing of the global migrate or dry residue of extracts is not usually practicable with paper as its quantity is determined too much by released fibres, with the result that it has little or no significance with regard to the substance in question (e.g. a paper finishing agent). For this reason, the extracts and migrates are tested specifically for the above-mentioned substances.

2 Fundamental considerations and methods for determining the transfer of substances from paper

The different approaches for determining the transfer of substances from a paper to the food it comes in contact with are outlined below.

Migration to the food is the fundamental aspect which always applies when providing evidence of safety for the use applied for. Models (conventions) have been developed as this is often difficult to determine. These are usually conservative and because they can underestimate the contamination that actually occurs in certain situations, they should be used with caution. Conversely, the models can also greatly overestimate actual contamination. In this case, the applicant is at liberty to derive a more realistic contamination level.

As a basic principle, applicants should assume in their considerations a ratio of the paper surface to the food mass which takes into account the typical and/or normally predictable (worst case) applications. If use is made of the convention which applies in the field of plastics that 6 dm^2 of packaging come into contact with 1 kg of food, this should be proven by means of application examples and the application limited accordingly where necessary for less favourable ratios. Typical and/or normally predictable (worst case) applications should also be used where the grammage is concerned. If there are no points of reference for the actual application, a grammage of 300 g/m^2 should be assumed.

All assumptions made must be justified in a comprehensible manner.

The model systems described below form a hierarchy. If the estimate of exposure based on one of the models arrives at results for which the extent of submitted toxicological data complies with the requirements of the Note for Guidance the following tests can be omitted.

2.1 Theoretical calculation on the assumption of a complete transfer based on the quantity used in the process

In this case it is assumed that the entire quantity of the substances used in the process remains in/on the paper during manufacture and is transferred to the food. The analytically determined composition of the commercial product should be used as the basis here. This calculation is mandatory, regardless of the further determinations carried out.

2.2 Theoretical calculation on the assumption of a complete transfer based on the content in the paper

Typically for an application a test paper (usually 300g/m^2 or conversion of the results to 300g/m^2, cf. 2) of commercial composition and with the maximum required amount of the requested substance has to be produced. An accumulation of the active substance in the process water is to be considered if applicable. The amount of substances in the test paper must be determined analytically. In case of use of extraction methods the completeness of the extraction has to be confirmed. Thereafter, the content in the food is calculated based on the assumption of complete transfer from the paper.

2.3 Migration measurement

2.3.1. Migration measurement in simulants

The starting point for these calculations is the results of the determination of substances in migration solutions obtained with the finished paper (cf. 3.2-3.4):

- Conditions based on the provisions of Annex V, Chapter 2, of Regulation (EU) No. 10/2011
- Determination of the transfer from paper and board using modified polyphenylene oxide (MPPO) as a simulant (DIN EN 14338)

Migrates as defined by this document can also be prepared by the following conventional methods:

- Cold water extract according to DIN EN 645
- Hot water extract according to DIN EN 647
- Organic solvent extract according to DIN EN 15519

It is assumed that the transfer measured in the simulant is the same as the transfer to the food.

The solubility of the substance in the simulants used must also be listed in the application under 2.1.4 *Solubility*.

According to the current level of available scientific knowledge, the use of simulants can be limited or adapted in line with the physical-chemical properties of the substances to be determined.

2.3.2 Migration measurements in foodstuffs

This approach is necessary if there is doubt that it is possible to determine actual migration with simulants. The test foods and contact conditions should be selected in accordance with the scientific knowledge.

2.4 Expression of results

The results according to 2.1 to 2.3 are to be presented in a table and are to be discussed for plausibility (fate and behaviour of the active substance in the process water, irreversible binding to the paper matrix).
A template for the summary is available at:
http://www.bfr.bund.de/cm/343/vorlage_stoffuebersicht.xls.

3 Test conditions for the individual recommendations

The procedure outlined in 2., above, can be implemented by means of one of the test schemes listed below.

3.1 Preliminary remark on extraction and/or migration tests (applies to all recommendations listed below)

The applicant is responsible for selecting the suitable test conditions. To do so, it is necessary to know the properties (e.g. solubility, polarity or volatility) of the substances which could potentially be transferred and have information on the intended use of the finished paper. With amphiphilic substances, it should also be taken into account when selecting the extraction solvent or simulants that substances with an emulsifying effect occuring in foods have an influence on the extent of the transfers. Papers are often in contact with dry food. In that case the transfer may occur by evaporation and re-condensation in the food for what a low vapor pressure is already sufficient. In the selection of test conditions the possibilty of such a gas phase transfer has to be taken into consideration also at lower temperatures.

The test conditions must reflect the worst case conditions that can occur in actual practice. In all extraction and migration experiments a paper which was manufactured with the maximum intended quantity and which was not pre-treated is to be used. In addition, it may also be reasonable to include into the tests a paper which was manufactured without the requested substance in order to recognise which substances or signals do not originate from the substance for which the application is made but from the paper itself.

On the basis of the considerations mentioned above, the below-listed extraction agents/ simulants/test foods should merely be regarded as examples for which the applicant has to check whether they actually do constitute the worst case or whether an alternative should be chosen. In that solubility observations and recovery experiments are usually helpful.

Provided that it can be proven that the transfer rates measured with one simulant or extraction solvent constitute the worst case, sole testing with this one is sufficient.

3.2 Papers pursuant to Recommendation XXXVI

Migration calculation based on conventional methods:

- Cold water extract according to DIN EN 645

- Organic solvent extract (isooctane/95 % ethanol) in accordance with DIN EN 15519
  or
- Migration tests according to the provisions of Regulation (EU) No. 10/2011
- where appropriate determination of the transfer from paper and board using modified polyphenylene oxide (MPPO) as a simulant (DIN EN 14338)

3.3 Paper pursuant to Recommendation XXXVI/1

Migration calculation based on conventional methods:

- Hot water extract according to DIN EN 647
- Organic solvent extract with isooctane/95 % ethanol, in accordance with DIN EN 15519 (only when required due to the polarity of the food)

Migration tests are not planned in this case as the papers are extracted in the course of their utilisation.

3.4 Paper pursuant to Recommendation XXXVI/2

Migration calculation based on conventional methods:

- Hot water extract (DIN EN 647)
- Organic solvent extract (isooctane/95 % ethanol, in accordance with DIN EN 15519)

Or

Migration tests:

- 2 hrs at 220 °C using modified polyphenylene oxide (MPPO) as a simulant (DIN EN 14338)

alternatively:

- 2 hrs at 220 °C with test dough in accordance with DIN 10955, Section 11.2.5.4
- For use of paper and board in microwave applications, only, migration test conditions are 30 min at 150 °C.

Thermostability:

Thermostability testing is required for substances that are to be included in Recommendation XXXVI/2. The requested substance should be tested (without paper matrix) as follows by means of thermogravimetry: a suitable sample quantity is heated to 220 °C and left at this temperature for 2 hours. The sample is then heated further to 250 °C and kept there for 10 minutes. For microwave applications the sample is heated to 150 °C and left at this temperature for 30 minutes. 10 °C per minute should be selected as the heating rate; the test is conducted under the influence of atmospheric oxygen (no nitrogen purging). The accuracy of the method used has to be stated. The thermogravimetric test must include the decomposition temperature and it may be necessary to heat to a temperature higher than 250 °C. Released volatile products must be specified by quality and quantity. If the sample shows signs of decomposition before 250 °C have been reached, the decomposition products that occur must

also be determined qualitively and quantitively and included in the application. Suitable methods are e.g. thermal desorption and purge and trap. Possible reactions with food ingredients in accordance with 2.2.7 of the Note for Guidance have to be discussed.

4 Additional notes

4.1 Applications for perfluorinated substances

The following should be observed when applying for the inclusion of substances which contain fluorine atoms: the atomic radius of fluorine is considerably smaller than that of hydrogen. Because of this, molecules in which several hydrogen atoms have been replaced by fluorine can have a considerably higher molecular weight without a change in the actual volume of the molecule. In addition, perfluorination increases the volatility and thereby the susceptibility to migration of substances of this kind. The assumption that substances with a molecular weight of > 1000 Daltons can no longer be resorbed in the gastrointestinal tract due to the size of their molecules and therefore do not pose a toxicological risk cannot be made for this reason. With regard to molecular weight distribution, it must therefore be specified in applications for polymeric perfluorinated substances how large the proportion of molecules up to 1500 Daltons is.

4.2 Screening

Impurities in the requested substance caused by the manufacturing process must be recorded analytically and characterised both qualitatively and quantitatively. Additional reaction and breakdown products can be formed when the substance is used for its intended purpose in the paper production process. These can be derived from the chemistry of the application process, but have to be verified and quantified analytically and tested for completeness. Screening is required for this reason. An analytical approach which is as comprehensive as possible and which covers all relevant components should be used for screening.

4.3 Analytical method for official control

As described in 5.1.8 and 6.5 of the Note for Guidance, a method for determining the substance in finished paper must be submitted which can be used by the official control authorities.

4.4 Traceability, raw data

All material specifications must be clearly documented. This applies in particular for analytical data (e.g. on transfer of substances). The applicant shall make available any raw data (peak areas, etc.). For chromatographic methods, chromatograms, at least typical ones (standards, samples, blanks), must be supplied in addition. Attention must be paid to the good readability of all relevant details.