Packaging Material Toxicity and Aseptic Packaging System for Food and Pharmaceuticals

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Why is Toxicology Approval required?

Ensures that materials in contact with foods are safe and will not transfer undesirable substances to the foods.
Approval must be obtained for

Materials in contact with food e.g.

- Packaging materials (direct or indirect)
- Construction materials e.g. conveyor belts, mixing vessels
- Foods contact promotional items e.g. ceramic mugs, kitchen utensils
Food Contact Approval

Foods contact materials should be considered in their entirety i.e.

- The material components
- The foodstuff in contact
- The conditions of use both within the supply chain and by the consumer
Evidence required that the material is **suitable for the intended use** when products are heated (>20°C) in contact with a good contact material during:

- Processing and filling
- Storage / transit
- Preparation by the consumer
Recycled / recovered Board
The use of recycled board is not recommended

Recycled Plastics
- Virgin materials
- Scrap from the immediate manufacturing process
- General factory waste and plastics from unsorted domestic waste

Glass, Steel, Tinplate and Aluminium
Inks & non-food contact lacquers/Varnishes

Rules

- Acceptable for incidental food contact
- Conform to one of the following guidelines for the manufacture of printing inks for foods packaging:
  - EuPIA / CEPE Exclusion list (European Printing Inks Association) [www.cepe.org](http://www.cepe.org)
  - Japan Printing Ink Makers Association, Voluntary Regulation Concerning Printing Inks for Food Packaging Materials (Negative List Regulation)

NOT suitable for direct contact with food!
Migration

Definition

Movement of chemicals and additives from the food contact material into the food.

Legislation on food contact materials

- Limit the migrations of substances from the food contact material into the foodstuffs
- Substances with toxic properties need to be controlled in order to prevent the concentration in the foodstuffs reaching a level which is injurious to human health.
In the USA the use of monomers and additives in plastic for contact with food is governed by FDA Title 21 Code of Federal Regulations part 177.

FDA 21 CFR specifies requirements for extraction / migration testing including test conditions and extraction / migration limits.

- Plastic must undergo appropriate migration testing

- All – plastic constructions
  - extended to plastic coatings and laminated materials

- Give approved list of monomers (330)
  - Specific migration limits or residual amount limits
The compound which have been set with a non-detectable SML, are those considered to have significant hazardous properties.

Some monomers have been given limits on residual levels in the plastic. These limits are maximum permitted quantities of the residual substance in the material or article (QM) and are mainly applied to monomers that are unstable in the presence of water where a specific migration limit is inappropriate. QM levels are typically set at 1 mg/kg plastic (1ppm)
4 model foodstuffs specified as food simulants; distilled water, 3% acetic acid, 10% ethanol and olive oil

Appropriate food simulant selected for each application (EC Directives 85/572/EEC and 97/48/EC)

Appropriate test conditions selected for each application (EC Directives 97/48/EC, 93/8/EEC and 82/711/EEC)
<table>
<thead>
<tr>
<th>Simulant</th>
<th>Simulates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Simulant A</td>
<td>Distilled water or water of equivalent quality</td>
</tr>
<tr>
<td>Simulant B</td>
<td>3% Acetic acid (w/v) in aqueous solution</td>
</tr>
<tr>
<td>Simulant C</td>
<td>10% Ethanol (v/v) in aqueous solution</td>
</tr>
<tr>
<td>Simulant C</td>
<td>50% Ethanol (v/v in aqueous solution)</td>
</tr>
<tr>
<td>Simulant D</td>
<td>Rectified olive oil (or HB307, a mixture of synthetic triglycerides)</td>
</tr>
</tbody>
</table>
Food Simulant D
(Rectified olive oil)

- Most aggressive simulant
- Greater extractive capacity than most foods
  - Reduction factor e.g. margarine, butter – reduction factor is 2

Alternatives to Simulant D
No use of fatty food simulants for technical reasons:
- 95% ethanol
- ISO – octane

The most conditions should be equivalent to / more severe (compared with simulant D)
<table>
<thead>
<tr>
<th>Ref. no.</th>
<th>Description of foodstuffs</th>
<th>Simulants to be used</th>
</tr>
</thead>
<tbody>
<tr>
<td>02.03</td>
<td>Cereal flour and meal</td>
<td></td>
</tr>
<tr>
<td>02.04</td>
<td>Macaroni, spaghetti and similar products</td>
<td></td>
</tr>
<tr>
<td>02.05</td>
<td>Pastry, biscuits, cakes and other bakers wares, dry:</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A. With fatty substances on the surface</td>
<td>X/5</td>
</tr>
<tr>
<td></td>
<td>B. Other</td>
<td></td>
</tr>
<tr>
<td>02.06</td>
<td>Pastry, cakes and other bakers wares, fresh:</td>
<td>X</td>
</tr>
<tr>
<td></td>
<td>A. With fatty substances on the surface</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B. Other</td>
<td></td>
</tr>
<tr>
<td>03</td>
<td>Chocolate, sugar and products thereof</td>
<td></td>
</tr>
<tr>
<td></td>
<td>confectionery products</td>
<td></td>
</tr>
<tr>
<td>03.01</td>
<td>Chocolate, Chocolate-coated products, substitutes and products coated with substitutes.</td>
<td>X/5</td>
</tr>
</tbody>
</table>
**Test Conditions**

Must represent food contact conditions for normal and foreseeable use

<table>
<thead>
<tr>
<th>Contact Time</th>
<th>Test conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>$t \leq 0.5\text{hrs}$</td>
<td>0.5hrs</td>
</tr>
<tr>
<td>0.5hrs &lt; $t \leq 1\text{hr}$</td>
<td>1 hr</td>
</tr>
<tr>
<td>1 hr &lt; $t \leq 2\text{hrs}$</td>
<td>2 hrs</td>
</tr>
<tr>
<td>2 hrs &lt; $t \leq 4\text{hrs}$</td>
<td>4 hrs</td>
</tr>
<tr>
<td>4 hrs &lt; $t \leq 24\text{hrs}$</td>
<td>24 hrs</td>
</tr>
<tr>
<td>$t &gt; 24\text{hrs}$</td>
<td>10 days</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Contact Temperature</th>
<th>Test Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T &lt; 5\text{Deg C}$</td>
<td>5\text{Deg C}</td>
</tr>
<tr>
<td>5\text{Deg C} &lt; $T \leq 20\text{Deg C}$</td>
<td>20\text{Deg C}</td>
</tr>
<tr>
<td>20\text{Deg C} &lt; $T \leq 40\text{Deg C}$</td>
<td>40\text{Deg C}</td>
</tr>
<tr>
<td>40\text{Deg C} &lt; $T \leq 70\text{Deg C}$</td>
<td>70\text{Deg C}</td>
</tr>
<tr>
<td>70\text{Deg C} &lt; $T \leq 100\text{Deg C}$</td>
<td>100\text{Deg C} or reflux temperature</td>
</tr>
<tr>
<td>100\text{Deg C} &lt; $T \leq 121\text{Deg C}$</td>
<td>121\text{Deg C}*</td>
</tr>
<tr>
<td>121\text{Deg C} &lt; $T \leq 130\text{Deg C}$</td>
<td>130\text{Deg C}*</td>
</tr>
<tr>
<td>130\text{Deg C} &lt; $T \leq 150\text{Deg C}$</td>
<td>150\text{Deg C}*</td>
</tr>
<tr>
<td>$T &gt; 150\text{Deg C}$</td>
<td>175\text{Deg C}*</td>
</tr>
</tbody>
</table>

* This temperature shall be used only for simulant D. For simulants A, B or C the test may be replaced by a test at 100\text{Deg C} or at reflux temperature for a duration of four times the time selected.
### Test conditions

- **Long Term storage / shelf live**: 10 days at 40 Deg C
- **Ice cream**: 10 days at 5 Deg C
- **Soup (consumer)**: 1 hour at 100 Deg C + 10 days at 40 Deg C

Testing done with 1 sample – multiple conditions after each other

*Test conditions must represent actual conditions of use*
## Frequency of re-testing

<table>
<thead>
<tr>
<th>Migration Result</th>
<th>Frequency of Re-testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;3.33 mg/dm² or &lt;20 mg/kg</td>
<td>Test results OK. Retest once per annum on the product or a very similar product from the same supplier</td>
</tr>
<tr>
<td>3.33 – 6.66 mg/dm² 20 – 40 mg/kg</td>
<td>Test immediately on a different samples to check result is repeatedly acceptable and then re-test at least once per year</td>
</tr>
<tr>
<td>&gt;6.66 mg/dm² or . 40 mg/kg</td>
<td>Test immediately of different samples until the results are statistically acceptable. Test each batch until results can be shown to be a true representation.</td>
</tr>
</tbody>
</table>
Exceptions to Migration testing (2)

Migration testing is not required for

- dry non–fatty (surface) products
- frozen products*
- Package not 100% plastic (i.e. wrapper: PP, paper, alum)
## Acceptable Regulations and Standards for Food Contact Materials

The following regulations and standards are recognised by SEAC as acceptable support for food contact materials:

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>Standards</th>
</tr>
</thead>
<tbody>
<tr>
<td>FDA 21 CFR 175.105</td>
<td>Adhesives</td>
</tr>
<tr>
<td>FDA 21 CFR 175.300</td>
<td>Resinous &amp; polymeric coatings</td>
</tr>
<tr>
<td>2002/72/EU and amendments (adopted into member state legislation)</td>
<td>Commission Directive 2002/72/EC relating to plastic materials and articles intended to come into contact with foodstuffs</td>
</tr>
<tr>
<td>BfR XIV</td>
<td>Plastics dispersions</td>
</tr>
<tr>
<td>BfR XXVIII</td>
<td>Cross-Linked Polyurethanes as Adhesive Layers for Food Packaging Materials</td>
</tr>
<tr>
<td>Australia and New Zealand regulations relating to Food Contact Materials</td>
<td></td>
</tr>
<tr>
<td>INDIA regulations relating to Food Contact Materials</td>
<td></td>
</tr>
<tr>
<td>JAPANESE FOOD SANITATION LAWS relating to Food Contact Materials</td>
<td></td>
</tr>
<tr>
<td>MERCOSUR regulations relating to Food Contact Materials</td>
<td></td>
</tr>
</tbody>
</table>

**Aluminium**

- It is a food contact alloy under the terms of reference in EN573
- Chemical composition and form of aluminium and aluminium alloy wrought
Conditions for food grade paperboards as per US FDA

Migration levels should be less than 0.5mg/sq.in with the reagent prescribed in US FDA 176.170.

Only the list of chemicals listed in 21 CFR 176.170 to be used in the manufacture of paper and boards

Optical brightening agents are not allowed for contact with liquid food.
Types of Boards and Structure

- **Top Coating**: 8-15 GSM
- **Pre Coating**: 7-10 GSM
- **Sizing**: 2 GSM
- **Top Layer**: 50-70 GSM
- **Filler Layer**:
- **Bottom Layer**: 30-50 GSM
- **Back Coating**: 2-10 GSM

<table>
<thead>
<tr>
<th>Layer</th>
<th>White Lined Chip Board</th>
<th>Folding Box Board</th>
<th>Solid Bleached Sulphate Board</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2,7</td>
<td>Coating Pigments &amp; Chemicals</td>
<td>Coating Pigments &amp; Chemicals</td>
<td>Coating Pigments &amp; Chemicals</td>
</tr>
<tr>
<td>3</td>
<td>Starch</td>
<td>Starch</td>
<td>Starch</td>
</tr>
<tr>
<td>4</td>
<td>Chemical Pulp</td>
<td>Chemical Pulp</td>
<td>Chemical Pulp</td>
</tr>
<tr>
<td>5</td>
<td>Waste Paper</td>
<td>Mechanical Pulp</td>
<td>Chemical Pulp</td>
</tr>
<tr>
<td>6</td>
<td>Waste Paper</td>
<td>Chemical Pulp</td>
<td>Chemical Pulp</td>
</tr>
</tbody>
</table>
Paper and Board for food contact as per BfR XXXVI

- There must not be any migration which affects the taste or smell.
- The finished paper/board must not contain more than 0.15mg/kg pentachlorophenol.
- There is no transfer of metal ions to foods: hence quantum of maximum heavy metals presence in paperboard should be (not applicable for dry and non fatty food)
  - Cadmium 0.5 ug/gram
  - Lead 3 ug/gram
  - Mercury 0.3 ug/gram
Paper and Board for food contact as per BfR XXXVI

• Azo dyes must not be used in the manufacture of food contact paper or boards.

• The boards must not have any preserving effect on the food stuffs with which they come in contact.

• Only chemicals and ingredients listed in BfR XXXVI as of 1.06.2007 can be used.
Steps to avoid odour in paperboards

- Careful selection of mechanical and chemical fibre
- Coating material subjected to stringent specification and control to reduce tainting risks
- Steps are taken to eliminate biological activity in the machine system
- Storing paperboard in an odour free area.
- Do not use recycled base boards for aroma sensitive products
Recycled Content in Paperboard for food packaging

- Paperboard manufactured with recycled fibres may contain Diisopropynaphthalene (DIPN).
- Fat containing food stuffs and food stuffs with large surface like rice, pasta with egg, chocolate coated biscuits, nut biscuits egg biscuits can take up DIPN in high proportions.
- Additional intermediate packaging required in the above cases.
- For direct contact only mill virgin broke and returned material of virgin grade boards to be used.
**Other guidelines for Food packaging**

- Paper/board manufactured with ECF pulp recommended as they do not contain detectable levels of Dioxins.
- Heavy metals like arsenic, antimony, chromium to be less than 20 PPM each.
Other guidelines for Food packaging

EU Standard - Food allergens a list of 66 chemicals listed in

Microbial Load:
- < 1000 CFU for general packaging
- < 250 CFU for aseptic packaging
Testing Taint and Odour

- Personal Testing
  - Triangle test (DIN 10951)
  - Pair test (DIN 10954)
  - Robinson test (DIN 10955)

- Gas Chromatography

- Mass Spectrometry
Statutory support required for packaging material

- BOARD
  - FDA 21CFR 176.170 and/or 176.180
  - BfR (BgVV) XXXVI and/or XXXVI(i)
  - Australia and New Zealand regulations relating to Food Contact Materials
  - INDIA regulations relating to Food Contact Materials
  - JAPANESE FOOD SANITATION LAWS relating to Food Contact Materials
  - MERCOSUR regulations relating to Food Contact Materials.
Statutory support required for packaging material

• PAPER
  - FDA 21CFR 176.170, 176.180
  - BfR (BgVV) XXXVI, XXXVI/1
  - Australia and New Zealand regulations relating to Food Contact Materials
  - INDIA regulations relating to Food Contact Materials
  - JAPANESE FOOD SANITATION LAWS relating to Food Contact Materials
  - MERCOSUR regulations relating to Food Contact Materials
Sterilization is the finite method for microbial control and can be achieved either by sterilizing each component (product and packaging material) followed by assembly aseptic processing, or by a terminal sterilizing process which involves both product and pack. Both processes are common to the industry.

Examples
Neutraceuticals / Medical Foods / Infusions – Aseptic processing
Medical Devices – Terminal Sterilization
Achieve Sterility of the package and packaging closure before being filled thru:

- Wet heat or wet steam to inactivate spore forming bacteria. Hot air, super heated steam can be used but it does not transfer its heat to the packaging material very efficiently. Also most plastics are heat sensitive.
Aseptic Package Sterilization

Chemical sterilant along with modest heat to inactivate the bacterial spores. Bacterial spores are the most resistant forms of bacteria to all sterilization processes. Approved sterilants are Hydrogen Peroxide and Per acetic Acid.
Aseptic Package Sterilization

- **Irradiation Processes:**

Used for aseptic bulk container sterilization for large volume bag in box products. The bag in a box product is assembled into its final form and sealed prior to radiation exposure. The radiation sterilizes the bag and its seal maintains sterility until it is opened and filled in the sterile zone of an aseptic packaging operation. Irradiation can be used to sterilize heat sensitive packaging materials. Types of irradiation include ionizing radiation (beta or gamma rays) infrared radiation and ultraviolet radiation.
Aseptic packaging systems combine the product, package and closure in a sterile environment to manufacture a product. Standard systems are:

- Fill and Seal
- Erect Fill and Seal
- Form Fill and Seal
- Thermoform fill and Seal
- Blow mould fill and Seal
- Bulk Storage and Packaging
The process relies on heat of extrusion to sterilize the container.

Plastic is extruded and blow moulded using sterile air to form the plastic into the final container shape in the blow moulding cavity / die.

The package may move immediately into the sterile zone for filling and closing or resealed using excess plastic.

This second bottle producing method is used in a process called extrusion blow moulding.

In this process a parison or melted plastics take emerges from the extruder and placed in the centre of the bottle producing mould.
The bottle which is sterile on the inside is then introduced to a sterilization system that desterilizes the outside of the container or the neck of the container as it enters the sterile zone where it is opened filled and sealed. The dome covering the neck of the bottle to produce a bottle finish compatible with the closure used for bottle. It may also receive a heat sealed membrane or lid stock prior to application of closure.
Converting For Sterilization

Important Parameters to be monitored

- Cytotoxicity of packaging materials.
- Material characteristics pre-post sterilization of packaging materials.
- Five year ageing comparison of finished packs
- Physical properties of packaging materials specially with reference to migration / leading and absorption.
- Heat seal curves – Prototype labs for market ready samples
- Root cause investigation – microbiological lab

Convertor could help by providing comprehensive technical data reports in advanced validation.
Thank you